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SAMPLING IN IRON ORE OPERATIONS

EVALUATION AND OPTIMISATION OF SAMPLING SYSTEMS
TO REDUCE TOTAL MEASUREMENT VARIABILITY

BY
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Sampling in iron ore operations

Evaluation and optimisation of sampling systems to reduce total measurement variability

Ph.D. Dissertation

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Till min bästa vän och stora kärlek Calle
Till Lo and Titus

Ni är mitt allt

Only those who risk going too far
can possibly know how far one can go

T.S. Eliot

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List of publications

Paper I *(published)*
Engström, K. (2017) A comprehensive literature review reflecting fifteen years of debate regarding the representativity of reverse circulation vs blast hole drill sampling. TOS Forum, 7:36-46. DOI: 10.1255/tosf.99

Paper II *(published)*
Engström, K. and Esbensen, K.H. (2017) Blasthole sampling (replicate and variographic experiments) in LKAB open pit iron ore mines: fit-for-purpose representativity? In: Dominy SC, Esbensen KH, editors. WCSB 2017. In Proceedings of the 8th World Conference on Sampling and Blending; May 9-11; Perth; AusIMM. pp 85-96.

Paper III *(published)*
Engström, K. and Esbensen, K.H. (2017) Optimal grade control sampling practice in open pit mining – A full-scale Blast Hole vs Reverse Circulation variographic experiment. Applied Earth Science - Transactions of the Institutions of Mining and Metallurgy: Section B, 126(4):176-187. DOI: 10.1080/03717453.2017.1414104.

Paper IV *(revised and resubmitted)*
Engström, K., Olausson, L. and Esbensen, K.H. (2018) Experimental evaluation of surface water sampling variability for environmental monitoring in iron ore operations using concepts from the theory of sampling (TOS).
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Paper V *(published)*
Engström, K. and Esbensen, K.H. (2017) Evaluation of sampling systems in iron ore concentrating and pelletizing processes – Quantification of Total Sampling Error (TSE) vs. process variation. Minerals Engineering, 116:203-208. DOI: 10.1016/j.mineng.2017.07.008

Paper VI *(published)*
Engström, K., Mickelsson K-O., Töyrä, S. and Esbensen, K.H. (2018) Improvement of sampling for moisture content analysis in iron ore pellet feed – variographic characterization and on-line IR-analysis. In Proceedings of the 29th International Mineral Processing Congress; September 15-21; Moscow.

Paper VII *(published)*
Engström, K. and Esbensen, K.H. (2018) Variographic Assessment of Total Process Measurement System Performance for a Complete Ore-to-Shipping Value Chain. Minerals 8(7):310. DOI: 10.3390/min8070310.

Abstract

The aim of this PhD project was to optimise sampling and measurement systems in the iron ore operations of Luossavaara Kiirunavaara AB (publ) (LKAB), using concepts and methods from the Theory of Sampling (TOS). The main objectives included critical evaluation of current and alternative sampling and measurement systems, development of new approaches as well as implementation of selected improvements. An additional objective was to assess if variographic characterisation is applicable and effective for continuous control of measurement systems in iron ore mining and processing. LKAB has been mining iron ore in northern Sweden for over 125 years, specialising in iron ore pellet production since the 1950s. Producing highly refined iron ore products require reliable and precise grade-, process- and quality control based on representative sampling and measurement systems. The Theory of Sampling (TOS) is a complete theoretical foundation for enabling design and implementation of accurate and precise measurement systems in process industries and in other settings. TOS provides both practical solutions for correct (accurate) sample extraction and comprehensive empirical evaluation methods for assessment of total measurement system performance. In this study, TOS principles have been applied in the studies of important measurement systems in LKAB mining and mineral processing operations in Kiruna and Svappavaara. By evaluation of measurement system variability in relation to true process variability, comprehensive quality assessments have been performed, allowing to determine if measurement systems are fit-for-purpose or need improvement or replacement. Results from the PhD project show that empirical studies of material heterogeneity as well as measurement and process variability are critical success factors for evaluating if sampling and measurement processes are fit-for-purpose. The empirical results both support and transgress conventional TOS understandings regarding the representativity of e.g. open pit mine drill sampling, hammer samplers, manual grab sampling and slurry samplers. It has been shown that variographic characterisation, replication and duplicate experiments are powerful empirical approaches that can generate valuable information regarding measurement system performance. These methods provide numerical evidence that enables objective evaluation, in contrast to traditional visual inspection which is mainly based on engineering knowledge and assumptions. In summary, sampling processes conforming to TOS regulations are crucial to ensure that reliable measurement results are obtained and that valid decisions can be made. Furthermore, each sampling and measurement system needs to be evaluated in its specific application, with appropriate empirical methods, to allow for unbiased and objective determination of its performance.

Resume (Danish)

Dette Ph.d.-projekts formål er at optimere prøvetagnings- og målesystemer i forbindelse med processering af jernmalm ved Luossavaara Kiirunavaara AB (publ) (LKAB), ved hjælp af koncepter og metoder fra Theory of Sampling (TOS). De overliggende formål er: i) kritisk evaluering af de nuværende samt alternative metoder for prøvetagning og proces måling, ii) udvikling af nye tilgange og iii) implementering af udvalgte forbedringer. Desuden ønskes en vurdering af muligheden for anvendelse af variografisk karakterisering for kontinuerlig kontrol af målesystemer i forbindelse med jernmalm udvinding og processering. LKAB har udvundet jernmalm i det nordlige Sverige i over 125 år, og har siden 1950'erne specialiseret sig indenfor produktion af højtforarbejdede pellets. For at kunne producere sådanne produkter kræves der nøjagtig og pålidelig information og kontrol af koncentrationer, processer og kvalitet – som må basere sig på repræsentativ prøvetagning og måling. Theory of Sampling (TOS) er en fuldstændig teoretiske grundlag der bl.a. gør det muligt at designe og implementere nøjagtige og præcise industrielle proces målesystemer. TOS beskriver også praktiske løsninger for korrekt (retvisende) prøvetagning og har endvidere omfattende faciliteter for empirisk evaluering med henblik på overvågning og kontrol af målesystemernes effektivitet. I dette projekt er TOS principper blevet anvendt på de vigtigste industrielle målesystemer der er i anvendelse i Kiruna og Svappavaara. Gennem at sammenligne målesystem udsving med de underliggende processers sande variationer har det været muligt at gøre kvalitetsvurderinger som igen har gjort det muligt at gradere udvalgte målesystemer, enten som fit-for-purpose, eller at det er nødvendigt med forbedringer eller udskiftning. I denne sammenhæng påviser Ph.d. projektet at det er en kritisk succesfaktor at gennemføre empiriske karakteriseringer. De praktiske undersøgelsesresultater både understøtter, men er også i delvis modstrid med TOS konventionelle paradigmer vedrørende repræsentativ prøvetagning i dagbrud og industrielle processer vedrørende borekerner, hammer prøvetagere, manual enkelt-prøve udtagning (grab sampling) og slurry prøvetagning. Projektet har påvist at variografisk karakterisering, duplikat- og replikat eksperimenter udgør nødvendige empiriske tilgange for at kunne tilvejebringe den nødvendige information angående proces målesystemer. Disse evalueringsmetoder producerer kvantitativ, objektiv information i modsætning til konventionel visuel inspektion, som hovedsagelig er baseret på standard antagelser og erfaringer. Sammenfatningsvist konkluderer projektet at det er bydende nødvendigt at alle prøvetagnings processer er i fuld overensstemmelse med TOS' krav for at kritisk vigtige industrielle måleprocesser kan levere de pålidelige data, der er nødvendige for korrekt beslutningstagen. Det er et ufravigeligt krav at alle prøvetagnings- og målesystemer bliver evalueret i den relevante industrielle praksis, og med de relevante evalueringsmetoder, for at proces og produkt kvalitetskontrol kan dokumenteres at være objektiv og retvisende.

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PART 1

General overview

1. Introduction

This chapter presents an introduction to the PhD project, including a brief background of iron ore mining and processing at LKAB and the prerequisite of representative measurement systems for accurate and precise process control. The scope and objectives of the PhD project, i.e. the incentive for the case studies conducted and appended, are also presented.

1.1 Iron ore mining and processing

Iron is the fourth most abundant element in the earth's crust, where the majority is bound in silicate minerals. As the energy needed to separate the iron from these minerals is high, the economically viable ore deposits mined today mainly contain the rarer iron oxide minerals hematite and magnetite, for which the refining costs are much smaller.

In 1642, iron ore was first discovered in the northern parts of Sweden. The magnetite iron ores in Malmberget and Kiruna are mined by Luossavaara Kiirunavaara limited company (LKAB), founded in 1890. LKAB inaugurated their first pelletizing plant in 1955, and the core business today is to produce highly processed iron ore products that create added value to customers, Figure 1. The major and most important products for LKAB are sintered iron ore pellets for blast furnace and direct reduction steel-making (https://www.lkab.com/en/).

The process of sorting, concentrating and pelletizing the mined iron ore, to high quality sintered pellets, requires reliable process- and quality control throughout the complex value chain. One key feature for successful control of the refining processes is to utilize measurement systems that are fit-for-purpose for their intended application. As in many other process industries, representative sampling processes is one of the critical success factors for obtaining desired product quality that meet customer demands.

Figure 1. LKAB's newest and largest pelletizing plant in Kiruna (photo: Fredric Alm)

1.2 Sampling and process control

Samples are extracted, and measurements are performed, throughout the value chain of LKAB iron ore operations. Measurements stem from physical sample extraction combined with manual laboratory analysis, on-line sensor measurements as well as fully automated sampling and analysis facilities including robotic laboratories. A key factor in all process measurement systems is sampling, no matter if the measurements include physical sample extraction or pointing a sensor to an analytical volume in the process stream (Esbensen and Paasch-Mortensen 2010). Measurement systems are applied to all types of sub-processes and to vastly different materials in modern iron ore processing plants: from large pieces of broken ore and tailings on conveyor belts, to finely ground pellet concentrates and additives in slurry pipes and blender tanks, to filtered pellet feed, iron ore green pellets and sintered iron ore pellets on conveyor belts (Holmes 2010). A common denominator for process measurement systems is that analytical results are produced at regular intervals and are affected by variabilities stemming from both sampling of the heterogeneous materials involved, sample preparation and analysis. If variabilities from the measurement system become too large, the apparent process variation intended to be monitored, will not be observable with sufficient clarity and possibilities for successful process control diminishes.

Representative measurement systems are critical to optimize resource utilisation, maximize profitability, minimize financial risk and reach desired quality of final product (Holmes 2010). Representative measurement results can only be obtained when sampling and analyses are both sufficiently accurate and with acceptably high precision (i.e. low variability). This enable true process variations to be detected and managed with sufficient reliability. All sampling processes generate sampling errors that can only be eliminated, or minimized, through an understanding of how and why they occur (Esbensen and Paasch-Mortensen 2010, Gy 1998, Pitard 1993). The Theory of Sampling (TOS) presents a complete framework for how to eliminate the so-called *Incorrect Sampling Errors* (ISE) and minimise the magnitude of the remaining error effects called *Correct Sampling Errors* (CSE).

A powerful statistical tool for evaluation of the variability of both the process and measurement systems is the variographic approach. Variographic characterisation, in contrast to Statistical Process Control (SPC), enables quantification of the various sources of variability (including measurement uncertainty) as well as optimisation of sampling and analytical protocols (Minkkinen and Esbensen 2014, Minnitt and Esbensen 2017, Minnitt and Pitard 2008). Variographic characterisation can be applied to available process data, historical data and experimental data produced for specific evaluation of existing or alternative measurements systems.

1.3 Scope of present study

The aim of this PhD project was to optimise the sampling and measurement systems throughout the entire mining-to-final product value chain at LKAB. From blast hole drill sampling in the open pit mines, through process sampling in the refining processes, to customer sampling during ship loading of final products. One objective of the study was to use variograms to characterize all present sampling, measurement and process variabilities, to enable assessment of the complete measurement system performance. Variographic characterisation was applied to both existing process data and data from directed intervention experiments where further investigation was needed. Data analysis and interpretation focused on finding the root causes of variability at all levels in LKAB's iron ore operations and by employing concepts from TOS, necessary improvements could be recommended and implemented. An additional objective of the study was to assess if variographic characterisation is generally applicable and effective for continuous control of measurement systems in iron ore mining and processing.

This PhD project focused on producing state-of-the-art research conclusions concerning critical sampling and measurement systems in iron ore operations, to develop new approaches where necessary, as well as implementing practical improvements to existing measurement systems identified during the study. The study included measurement systems used in open pit mining, refining and pelletizing processes, as well as environmental surface water sampling at the LKAB iron ore operations in Kiruna and Svappavaara, Sweden. The seven appended papers links to the LKAB value chain as shown in Figure 2. Note that the underground mining operations in Kiruna and Malmberget are important parts of the LKAB value chain, producing approximately 85-90% of the crude ore for the processing plants. However, the grade control of the underground mining operations is not covered by this PhD study.

Figure 2. Link between iron ore operations in the LKAB value chain and the appended papers

2. Theoretical background

This chapter presents a comprehensive theoretical background regarding the LKAB iron ore operations and the Theory of Sampling, including in depth descriptions of both open pit mine drill sampling and process sampling. Furthermore, the applied methods for evaluation of measurement system performance are described. These theories have been used in the case studies of this PhD project, which were performed in order to reach the objectives presented in chapter 1.

2.1 LKAB Iron Ore operations

LKAB have been mining iron ore in the north of Sweden for more than 125 years. Since the mid-1950s, the competitive advantage has been production of sintered iron ore pellets for blast furnace and direct reduction steel-making, Figure 3, to create added value for customers. LKAB operates underground mines in Malmberget and Kiruna and open-pit mines in Svappavaara. Processing plants for refinement and pellet production are located at all three mine sites and the final products are transported by railway to the harbours in Luleå and Narvik. There are some differences between the processing plants in Kiruna and Svappavaara compared to Malmberget. As the case studies within this PhD project were performed in Kiruna and Svappavaara, these production processes are described in detail, while the operations in Malmberget are described in more general terms.

Figure 3. LKAB iron ore pellets for direct reduction steel making (photo: Fredric Alm)

LKAB is a small actor on the international iron-ore market but the second largest supplier of seaborne iron ore pellets. Europe is LKAB's largest export market, where customers demand high quality iron ore pellets for blast furnace steel-making (BF-pellets). The Middle East and North Africa (MENA), with good access to natural gas, is the second largest market with a strong demand for iron ore pellets for direct reduction steel-making (DR-pellets). Apart from BF- and DR-pellets, LKAB also produce industrial minerals, as for example high grade magnetite fines used for water purification and sound proofing, as well as heavy concrete ballast material (https://www.lkab.com/en/about-lkab/lkab-in-brief/).

Producing high quality products from the mined iron ore is a complex process and includes sorting, concentrating and pelletizing in industrial-scale processing plants. Correct process- and quality control in the complete value chain is critical for reaching the high product quality demanded by the customers. Environmental sampling, e.g. recipient surface water sampling, is also an essential activity for assessing, and take responsibility for the environmental impact of LKAB iron ore operations.

2.1.1 *Geological Setting and Mineralization*

Kiirunavaara in Kiruna is a high-grade iron ore deposit consisting mainly of magnetite and apatite, with an average grade of 63.8% iron and 0.4% phosphorus (estimated from the 3D resource model). The deposit is a sheet-like body, north to south striking and approximately 4 km to 4.5 km long, with a thickness of 50-100 m and a maximum of over 200 m in the northern part (Niiranen 2006). Common gangue minerals are apatite and minerals from the amphibole group. Significant SiO_2-bearing minerals include actinolite (amphibole), chlorite, phlogopite, titanite and also some quartz (Aupers 2014, Niiranen 2015, Nordstrand 2012).

The Leveäniemi deposit in Svappavaara is dominated by massive magnetite ore but in the central part of the deposit magnetite is largely altered to hematite (martite). Apatite, calcite and amphibole are the main gangue minerals in the deposit, while titanite, diopside, biotite and chlorite are less important (Bergman, Kubler and Martinsson 2001, Martinsson et al. 2016). The massive magnetite and magnetite veins/breccias in the trachyandesites and biotite schist are the main ore types mined in Leveäniemi. The Malmberget deposit is a high-grade iron ore deposit, consisting of approximately 20 separate ore bodies with an average iron grade of 51-61%. The majority of the ore bodies in the Malmberget ore field consist of magnetite, while a few in the western part host hematite or a mixture of magnetite and hematite. The surrounding rocks are felsic or mafic rocks of volcanic origin, often rich in biotite (e.g. Geijer 1930, Lund 2013, Martinsson and Virkkunen 2004).

2.1.2 *Underground mining*

LKAB operates underground mines in Kiruna and Malmberget. The underground mines host the main part of the mined ore reserves, contributing with approximately 85-90% of the ore for the processing plants. The ore is extracted using sub-level caving, Figure 4.

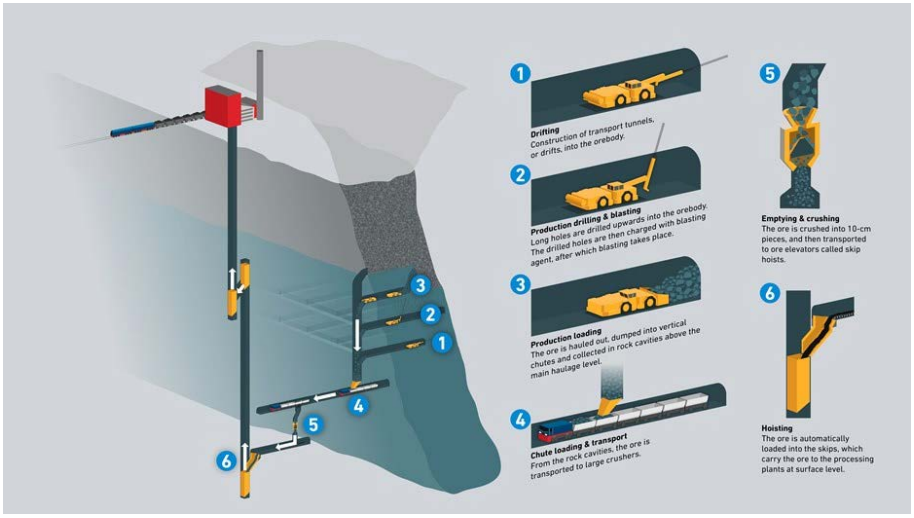

Figure 4. Underground mining at LKAB, using sub level caving (reprinted with permission: https://www.lkab.com/en/about-lkab/from-mine-to-port/mining/our-underground-mines/)

Sub-level caving is allowing maximum extraction from the ore body, with a high degree of safety, for the steep LKAB ore bodies. Mining includes drifting of underground tunnels, production drilling in an upward fan-shaped pattern followed by loading of explosives and blasting, which is performed every night. The ore is removed using loaders, tipped into vertical shafts that use gravity to move the ore to rock bins, and then transported to underground crushers by driverless trains (Kiruna) or large trucks (Malmberget). After crushing, the ore is conveyed to skip hoists lifting up to 40 tonnes of ore per skip, at a speed of 17 metres per second, to the sorting plants on the surface, (https://www.lkab.com/en/about-lkab/from-mine-to-port/mining/our-underground-mines/).

2.1.3 Open-pit mining

LKAB is currently (2018) operating one open-pit mine in Svappavaara, contributing to approximately 10-15% of the iron ore for the processing plants. The open-pit mining uses diamond drilling for initial exploration and block model development, while blast hole drilling is used for extended grade control and blasting. After blasting, loaders are used to load the ore on to large trucks that transport it to intermediate storage areas. (https://www.lkab.com/en/about-lkab/from-mine-to-port/mining/our-open-pit-mines/). The ore is blended to reach consistent quality into the sorting plant, which is critical for both optimized process performance and final product quality. Blast hole drill sampling for grade control is an important aspect for achieving desired iron grade in the ore blend for the sorting plant. Apart from iron grade, vanadium, manganese and silica grade are also important parameters in the open-pit mine grade control

2.1.4 Sorting

The sorting plant is the first plant of the ore processing value chain, Figure 5. The ore is crushed in cone crushers and waste rock is removed using magnetic separation. Sieving is used to divide the ore into three different size fractions and slightly different magnetic separation equipment is applied for the various sizes to increase grade and recovery. The largest size fractions are collected as grinding media for the autogenous grinding plant. Autogenous mills use larger rocks of ore as grinding media, to achieve compressive grinding of finer ore particles (https://www.lkab.com/en/about-lkab/from-mine-to-port/processing/sorting/).

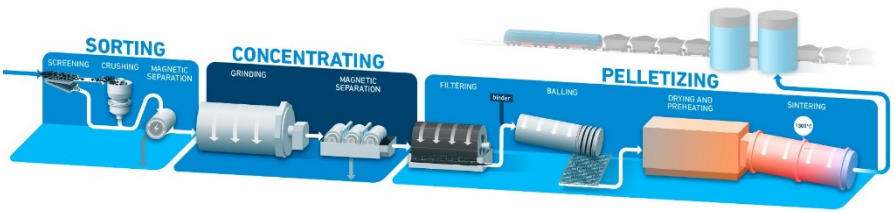

Figure 5. Schematic overview of the LKAB mineral processing value chain (reprinted with permission: https://www.lkab.com/en/about-lkab/from-mine-to-port/processing/)

Important measurement systems in the sorting plant address determination of iron grade of both incoming ore and outgoing final products in transport to the concentrating plants. Iron grade of the tailings is also measured to ensure that the magnetic separation is working satisfactorily, i.e. that material with high iron grade is not separated as waste and removed from the process.

2.1.5 Concentrating

The sorted and crushed material is conveyed to the concentrating plant, where it is mixed with water to form a slurry and finely ground in two or three stages. In between all grinding stages, magnetic separation, Figure 5, is used to remove gangue minerals containing impurities like silica, sodium, potassium and phosphorus. Magnetic separation increases the iron grade of the ore to around 68%. Flotation of the phosphorus gangue mineral apatite is used for further refinement and enrichment. In this process, a reagent is added to bind the apatite to small air bubbles that form a froth, which can be removed from the iron ore slurry. There is no flotation in the concentration plants in Malmberget as the phosphorus grade of the crude ore in the Malmberget mine is sufficiently low. (https://www.lkab.com/en/about-lkab/from-mine-to-port/processing/concentration/).

Important measurement systems in the concentrating plant concern determination of iron, silica and phosphorus grade in the different stages of the process. These results are used to control the performance of e.g. the magnetic separation for increasing iron grade and the flotation process for reduction of phosphorus grade. The size distribution and specific surface area are also measured throughout the grinding process as these are important parameters for successful balling in the subsequent pelletizing process.

2.1.6 Pelletizing

Various additives, depending on pellet type and specification, are mixed with the slurry, which is then filtered to reach the correct moisture content needed for balling of green pellets (non-sintered and non-oxidised iron ore pellets). The filtered iron ore concentrate is balled in large rotating drums with water and external binder as binding media, Figure 6. The green pellets from the balling drum are screened to separate the correct size fraction (10-16mm) for sintering. The under-size fraction is returned to the balling drum as seeds, while the over-size fraction is crushed and also returned to the drum. The circulating load is usually 1.2-2 times the amount of fresh feed inserted in the balling drum. A narrow size distribution of the finished green pellets is important for correct pellet quality, as high permeability is important for both oxidation and sintering of the iron ore pellets and in the subsequent steel making processes.

The green pellets are loaded on to the travelling grate, that passes through the drying and pre-heating zones where the oxidation from magnetite to hematite starts. The pellets are then transferred to the rotating kiln where they are sintered at a temperature of ≈1250°C, causing the iron ore particles to partially melt together, Figure 6. The LKAB pelletizing plants in Malmberget apply a straight grate process instead of the grate-kiln process used in Kiruna and Svappavaara. The oxidation from magnetite to hematite generate large amounts of

energy, resulting in a 60% reduction of the required energy input. Lastly, the temperature of the red-hot pellets is lowered in the cooler, before they are conveyed to the underground storage and subsequently loaded on to trains for further transportation to the harbours (https://www.lkab.com/en/about-lkab/from-mine-to-port/processing/pelletizing/).

Important measurement systems in the pelletizing plant include determination of moisture content of the filtered slurry and green pellets. Complete quality control of the final sintered iron ore pellets is also critically important and include several chemical, physical and metallurgical parameters, e.g. iron and silica grade, crushing strength, abrasion index, size distribution, reduction under load and degree of oxidation.

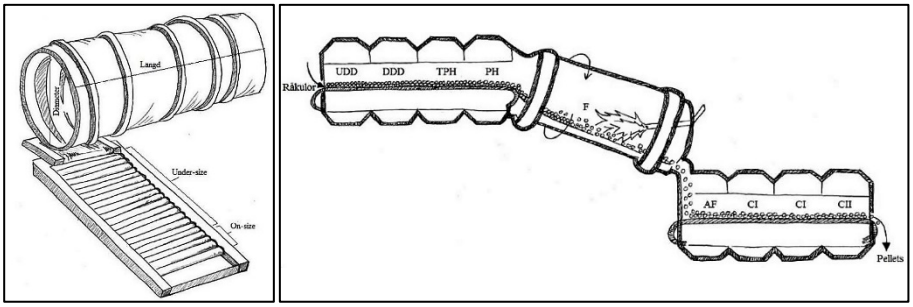

Figure 6. left: balling drum with subsequent roller screen, right: grate, kiln and cooler for oxidation and sintering of iron ore pellets (reprinted with permission: Björkvall 2018).

2.1.7 Transportation and shipping

The final products from LKAB operations in northern Sweden are transported by railway to the ports in Narvik and Luleå, located in northern Norway and Sweden respectively. Additives are transported back with returning trains, ensuring a sustainable and efficient transportation system. To optimise transportation efficiency, some of the world's strongest locomotives (IORE) are used to transport up to 6 800 tonnes of ore products in a single train, measuring 750 metres long and carries 68 wagons. When arriving at the harbour, the full train is unloaded in 30 minutes in advanced automated unloading stations, dropping the iron ore products in to underground silos. The harbour in Narvik is ice-free all year around and can accommodate large ocean-going vessels, Figure 7 (https://www.lkab.com/en/about-lkab/from-mine-to-port/transport/rail-transport/).

Two-thirds of LKAB's exports are shipped out of Narvik, where a second quay and ship loader were commissioned in 2016. The harbour is deep enough to accommodate ocean going vessels and single shiploads of up to 220'000 tons has been delivered to LKAB customers via Narvik harbour. One third of exports are shipped out of Luleå harbour, which has the advantage of a close distance to the European customers but with a more restricted depth compared to the Narvik harbour. In both harbours, state-of-the-art, highly automated sampling and measurement systems are in use to ensure representative analytical measurements of customer samples for contractual purposes (https://www.lkab.com/en/about-lkab/from-mine-to-port/transport/harbours/).

Figure 7. Narvik harbour with both ship loaders in operation (photo: Johanna Fogman)

2.1.8 LKAB process sampling and analysis

LKAB applies a wide range of methods for primary sampling, to collect samples from the production processes. Sample extraction ranges from manual grab sampling, cross belt hammer samplers, slurry shark fin samplers and spear sampling, to state-of-the-art linear cross stream samplers, complemented by measurements using various on-line sensors. Secondary sampling and sample preparation are performed automatically by in-line systems, as well as manually in sample preparation laboratories. The large variety of analyses related to process and quality control are performed both in fully automated measurement systems and through manual and mechanical analyses in the in-house laboratories.

The type of sample extraction used is largely dependent on the purpose and importance of the measurement system. I.e. for customer samples in the harbours (which are used for pricing of final products) state-of-the-art automated sampling systems, with linear cross stream samplers collecting a large number of increments, are in operation, Figure 8. For sub-processes needing frequent and precise control, automated sampling and measurement systems are normally applied. Three examples of completely automated in-line measurement systems at LKAB are:

i) Slurry sampling in the concentrating plants using shark fin or spear sampling. The primary sample is sub-sampled in two steps, then dried and transported by pneumatic dispatch to a robotic laboratory with automated grinding and briquetting. Lastly, the powder briquette is transported with a conveyor belt to the automated XRF analysis.
ii) In-line size analysis is also applied in the concentrating plant, using laser diffraction to determine the size distribution of the ground particles in the slurry. The percentage of <45µm particles from this analysis is used for continuous control of the grinding process.
iii) In-line size analysis of final iron ore pellets, in both pellet plants and harbours, employ linear cross stream samplers collecting increments every 3-10 minutes. Increments or composite samples are analysed by automated sieving machines up to every 4 minutes.

Some analyses are performed both on-line and in-line as described in the examples above. However, many analyses are also performed with a large variety of analytical equipment in the in-house laboratories. LKAB operates three sample preparation and physical analysis laboratories, two wet chemical and XRF laboratories, one metallurgical laboratory and one water laboratory for environmental samples. The majority of all extracted samples are analysed in the LKAB laboratories, while a small number of samples are sent for external analyses. This mainly occurs during peaks for environmental water sampling in summer and for some trace element analyses of water and ore that are not available in-house.

Figure 8. Two examples of linear cross stream samplers used in the pelletizing plants and Narvik harbour respectively. Left: Linear sampling bucket in maintenance position outside the ore stream. Right: Linear sampling bucket in rest position behind the belt discharge. (Photo: Jonas Gunnare)

2.1.9 LKAB environmental sampling and analysis

In contrast to the measurement systems applied for process and quality control, recipient surface water sampling is performed for environmental monitoring. These measurement systems enable assessment and control of the environmental impact of the LKAB iron ore operations. According to Swedish government regulations, the environmental impact by all industrial operators need to be assessed and understood by the operator that has the obligation to report truthfully to the authorities. The environmental permit that allow LKAB to continue to mine and process iron ore includes specific terms and the environmental impact of the operation must be controlled through self-monitoring programs. These normally include sampling protocols that specify sampling targets, frequencies and specification of analytical parameters and accreditation. The self-monitoring programs vary between the different LKAB mine sites, for example regarding sampling frequency, due to the individual environmental permits for each site.

The Swedish authorities provide guidance regarding which methods to apply for extraction of surface water samples. The recommendations are in practice describing a manual grab sampling approach applied to sampling targets in streams or lakes, Figure 9. These methods are also taught through certified commercial courses.

Figure 9. Surface water sampling in a recipient water stream for environmental monitoring.

2.1.10 Monitoring and control of sampling systems

The current monitoring and control of sampling systems at LKAB is mainly based on regular visual inspections. During maintenance shut downs of the processing plants, automated measurement systems are also subjected to regular and necessary maintenance and control. However, continuous statistical evaluation of sampling systems is not traditionally applied at LKAB. Evaluation of the performance of measurement systems, including characterisation of variability stemming from sampling, sub-sampling and sample preparation is thus not applied. In contrast, monitoring and control of the in-house laboratories follow comprehensive QA/QC programs, including analysis of Certified Reference Materials (CRM) and international inter-laboratory proficiency testing.

To allow for objective evaluation, as well as continuous monitoring and control of complete measurement systems (including sampling, sample preparation and analysis), statistical evaluation with the use of variographic characterisation, replication and duplicate experiments are versatile methods. Such approaches have previously been applied in a few studies at LKAB. However, a comprehensive initial evaluation as well as continuous monitoring and control of measurement system would be favourable. These issues played an important role in the initiation of the present Ph.D. project.

2.2 Theory of Sampling

The Theory of Sampling (TOS) was first described by Pierre Gy in the 1950s and since has been developed and extended to reach the level of a complete scientific theory, covering all aspects of sampling of particulate (and many other types of) materials (Pitard 1993). TOS includes comprehensive theoretical and mathematical definitions of sampling as well as practical applications to ensure representative sampling processes. For example, TOS i) provides a complete set of theoretical definitions of material heterogeneity and sampling variability, ii) describes correct increment delineation and sample extraction methods, iii) systemises nine types of sampling errors and iv) provides empirical methods for evaluation of sampling variability (Esbensen and Paasch-Mortensen 2010, Gy 1998, Minnitt and Esbensen 2017, Pitard 1993).

The practical purpose of sampling is to collect a small part, e.g. one gram for chemical analysis, of the lot to be assessed, e.g. a 220 000-ton shipload of iron ore pellets. The not so trivial aspect of this process is to ensure that the small analytical aliquot is representative of the total lot, e.g. that the measured iron grade is a reliable estimate of the average grade of the shipload of pellets. Hence, representative sampling processes are imperative, both for allowing correct grade determinations for commercial purposes and for valid process monitoring and control (Holmes 2010). There is no characteristic pertaining to the sample itself that can determine the status of representativity. Therefore, the only way to guarantee representative samples is to ensure that the complete sampling and measurement process, including primary sample extraction, sub-sampling, sample preparation and analysis, is representative (Esbensen and Paasch-Mortensen 2010). With comprehensive knowledge and application of TOS, all necessary tools for designing representative sampling processes are at hand (Gy 2004).

Sampling does not just involve one discrete operation but is a multi-stage process, Figure 10. The initial stage of the sampling process is the primary sample extraction from the lot. In addition, this process most often includes additional sampling stages (mass reduction/ sub-sampling). In between and/or after these sampling stages, sample preparation can be performed where needed. Preparation may include e.g. drying, crushing, grinding and/or dissolution. The sampling process results in one or several analytical aliquots with appropriate mass and properties for insertion in the analytical equipment.

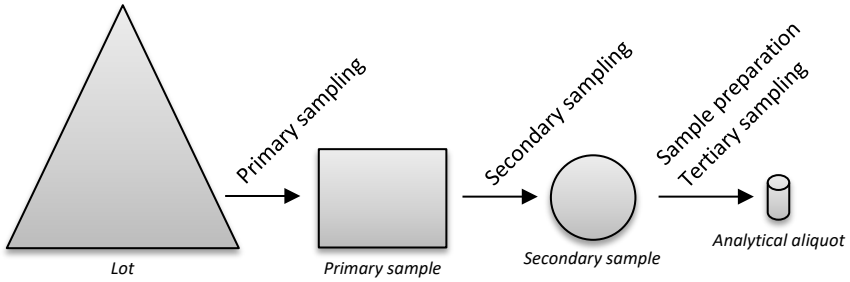

Figure 10. Sampling is a multi-stage process involving several sampling stages as well as necessary sample preparation.

2.2.1 Basic definitions and terms

For a comprehensive understanding of TOS, some important basic definitions and terms are described here (DS 3077 2013, Esbensen and Wagner 2014, Gy 1979, 1998, Pitard 1993).

Accuracy: Defined as the mean of the selection error. When the absolute value of the selection error is not larger than a predetermined threshold, the sample can be deemed accurate. There is an important distinction between an accurate and unbiased sample. A truly unbiased sample would imply that the mean of the selection error is zero, which is a limit case not possible in practice. The selection error is never strictly zero but may be negligible, leading to an accurate (but not unbiased) sample, with a *fit-for-purpose* accuracy.

Analytical aliquot: The final (often very small) sample that is inserted in the analytical equipment and used for quantifying the critical component.

Composite sample: A sample made up of several correctly extracted, individual increments.

Correct increment/sample selection: When all elements or particles of the lot have equal, non-zero probability of ending up in the sample. Correct increment/sample selection is essential in order to avoid an uncontrollable sampling bias.

Critical component: A chemical, physical or metallurgical component of which the proportion is to be measured. The component presenting most problems or restrictions should determine the parameters of the sampling process for all critical components to be measured of the lot. Can also be called *analyte.*

Fragment: Generic term for the smallest physically separable unit of the lot, e.g. particles, grains or fragments hereof.

Fundamental Sampling Principle (FSP): The basic prerequisite for a representative sampling process, stating that all elements or particles of the lot should have an equal and non-zero probability of being selected for the sample. Elements or particle not belonging to the lot should have zero probability of being sampled. FSP stipulates that the target for sampling must be the complete geometry/volume of the lot. The selected elements or particles should not be altered in any way after the increment/sample has been collected (see 2.2.3).

Heterogeneity: Homogeneity is defined as all elements of the lot are strictly identical, while heterogeneity is when all elements of the lot are not identical. All particulate materials in technology and industry are by this definition heterogeneous, affected by a combination of several kinds of heterogeneities (see 2.2.2).

Increment: A correctly delineated sampling unit that combined with other increments form a composite sample. In practical sampling, an increment is a correctly extracted group of spatially coherent fragments. The increment is extracted during a single operation of the sampling device.

Lot: The complete entity of original material being targeted for sampling, e.g. a shipload, process stream, 24h of produced iron ore pellets or an ore block in the open pit. The lot, also termed *sampling target*, refers to the physical characteristics and geometrical form (lot dimensionality) of the material, as well as to the total volume or mass.

Lot dimensionality: In practice, all lots are three dimensional. However, in practical applications, one or two of these dimensions can be significantly smaller than the third and can hence be regarded of secondary importance. The dimensionality of the lot can therefore also be defined as the effective number of dimensions of the material to be dealt with during practical sampling. TOS defines four possible lot dimensions, i.e. 0D, 1D, 2D and 3D lots, Figure 11.

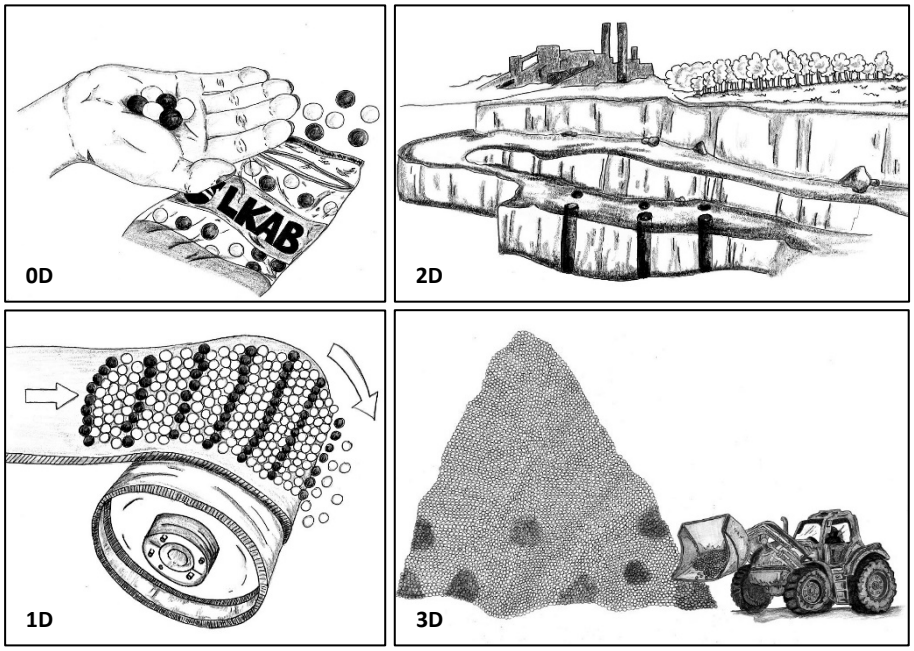

Figure 11. Visualisation of the four lot dimensionalities defined by TOS (printed with permission: © Maria Björkvall 2018).

The fewer lot dimensions, the easier it is to design and execute a representative primary sampling operation. The 0D lot is a special case where the material can be easily mixed and manipulated without undue effort, and therefore also sampled with complete correctness. This is usually the case for *small* lots, e.g. laboratory samples or analytical aliquots. 3D lots are (in contrast to 0D lots) *large* and therefore cannot be mixed or manipulated without invoking significant, often unacceptable efforts, time and resources. However, 3D lots can be sampled to fit-for-purpose representativity, using various implementation of composite sampling, manual or automated. Examples of 3D lots are railroad cars, trucks, large stockpiles and similar. In 2D lots, one dimension is limited in such a way that correct, practical increment extraction will always cover the complete extension of that dimension, e.g. a bench depth in an open pit mine (constant depth) or a geological formation (variable depth).

1D lots are preferred to maximise the possibilities of extracting representative samples. This either refers to a stationary elongated pile or a dynamic (moving) process stream, where two dimensions can be completely covered by extracting full cross-stream increments to form a composite sample. The third dimension is by definition substantially larger than the width and depth dimensions of a 1D lot. Appropriate sampling schemes can be used to cover the length of material in a representative fashion. Moving 1D transformations of 3D lots is the preferred, most often used approach, for acceptable sampling of large 3D lots in technology and industry. This is known as the principle of lot dimensionality transformation.

Precision: Defined as the variance of the selection error. Describes the reproducibility of a measurement and is a property of the variance of a given error. A measurement is deemed precise or reproducible when the variance of the selection error is lower than a predetermined threshold for a given purpose.

Representative sampling: A sample cannot by itself be deemed representative by any attribute. A representative sample can only occur as a result of a representative sampling process. Representative sampling is a multi-stage process where all fragments and/or increments have equal probability of being selected in every sampling stage to end up in the analytical aliquot. Representative sampling implies that the sampling process is both accurate and sufficiently precise.

Sample: A part of the lot, extracted using a representative sampling process. This means that the sample needs to be positioned, delimited and extracted in accordance to certain rules defined by TOS. A sample is normally obtained by extracting several increments to create a composite sample. A complete sampling procedure most often includes several stages, where all stages must comply to the rules defined by TOS.

Sampling bias: The sampling bias is defined as the mean of the sampling error. Non-accurate sampling processes will lead to an uncontrollable sampling bias. The sampling bias is different every time a set of increments (composite sampling) are extracted from the lot (due to the heterogeneity of the lot material) and it does not follow any statistical distribution. This means that it is impossible to quantify or correct for a sampling bias after the sample is extracted. The sampling bias stems from non-eliminated Incorrect Sampling Errors (ISE) and can only be sufficiently minimized by applying TOS correct sampling processes (see 2.2.3). The nature of the sampling bias is therefore completely different from that of an analytical bias, which often is systematic and can be corrected for.

Sampling protocol: A flowsheet or description of the sampling method, the frequency of sampling and the sequence of sampling and preparation stages applied to a lot of material. Resulting in one or several samples used for estimating critical components of the lot.

Sampling error: The relative sampling error, or selection error, occurs when the true, unknown grade of the lot is replaced by the estimation of the grade, e.g. the measured grade of a sample.

Specimen: A part of the lot obtained without respecting the rules for correct delimitation, extraction, preparation and weighting set by TOS. A specimen results from a biased sampling operation and can therefore never be accurate or representative of the lot.

2.2.2 *Heterogeneity*

Heterogeneity is a basic structural property of almost all materials in technology and industry and cannot be ignored in processing industries. Quality assessment of products, research and development or feasibility studies of process improvements all need to account for the heterogeneity of materials, e.g. when extracting samples, evaluating raw materials and designing or evaluating production processes. Process- and quality control as well as research and development in process industries are all concerned with estimates of critical constituents of one or another lot material. These estimates are always accompanied by uncertainties stemming from the sampling and analytical errors (Esbensen and Paasch-Mortensen 2010).

Sampling errors exist due to the inherent material heterogeneity present in almost all materials. There are a few types of materials that can be more or less exempt from this statement, including fine or pure chemicals, reagents, powders and liquid solutions. Examples include correctly prepared Certified Reference Materials (CRM) and liquid solutions within analytical chemistry applications, where the normal sampling problems are practically absent (Esbensen and Paasch-Mortensen 2010). However, most industrial sampling processes are applied to materials that are heterogeneous at one scale or another. The heterogeneity of a material is a direct consequence of the different size, shape, density, grade and other properties of the fragments and their spatial distribution in the lot (Pitard 1993). The inherent heterogeneity of the material can be discriminated at two scale levels, i.e. the Constitutional Heterogeneity (CH) and the Distributional Heterogeneity (DH).

CH is described by the intrinsic differences in physical and/or chemical properties between the fragments in the lot. As CH is dependent on the differences between fragments, at the current state of comminution, i.e. the current size distribution, mixing does not affect CH (Gy 1998). In the ideal case of homogeneity, i.e. where all fragments of the lot are strictly identical, CH would be zero. Even though this case can be theoretically defined, it is never present in natural material subjected to sampling (Gy 2004). The main concern in practical sampling is the heterogeneity between fragments rather than within fragments, and this is directly related to the grade of the critical component (a_i) of interest. To enable definition of CH of a lot, it is first necessary to define the heterogeneity (h_i) carried by a fragment in the lot (L). This is a function of the fragment weight (M_i), where a_L is the grade of the lot, $M_{\bar{i}}$ is the average fragment weight, N_F is the number of fragments in the lot and M_L is the mass of the lot, Equation 1 (Pitard 1993).

$$h_i = \left(\frac{a_i - a_L}{a_L}\right) \cdot \frac{M_i}{M_{\bar{i}}} = N_F \left(\frac{a_i - a_L}{a_L}\right) \cdot \frac{M_i}{M_L} \tag{1}$$

The Constitutional Heterogeneity of the lot (CH_L) is hence defined as a dimensionless variance of the heterogeneity carried by each fragment of the lot, Equation 2.

$$CH_L = s^2(h_i) = \frac{1}{N_F} \sum_i h_i^2 = N_F \sum_i \frac{(a_i - a_L)^2}{a_L^2} \cdot \frac{M_i^2}{M_L^2} \tag{2}$$

DH is described at a higher scale level as the differences between groups of fragments (e.g. increments) of the lot. DH is the main material characteristic to deal with during practical sampling as this necessarily involve extraction of increments from the lot. DH is defined through the heterogeneity carried by the effective groups of fragments (or similarly, carried by the average fragment in a group) in the lot (h_n), Equation 3 (Pitard 1993), where a_n is the grade of the group of fragments, M_n is the mass of the group of fragments, $\overline{M_n}$ is the average mass of the group of fragments, N_G is the number of groups and M_L is the mass of the lot.

$$h_n = \left(\frac{a_n - a_L}{a_L}\right) \cdot \frac{M_n}{\overline{M_n}} = N_G \left(\frac{a_n - a_L}{a_L}\right) \cdot \frac{M_n}{M_L} \tag{3}$$

The Distributional Heterogeneity of the lot (DH_L) is hence defined as a dimensionless variance of the heterogeneity carried by each group of fragments of the lot, Equation 4.

$$DH_L = s^2(h_n) = \frac{1}{N_G} \sum_n h_n^2 = N_G \sum_i \frac{(a_n - a_L)^2}{a_L^2} \cdot \frac{M_n^2}{M_L^2} \tag{4}$$

DH_L and CH_L are intimately linked as DH_L always is a fraction of CH_L. Therefore, by definition CH_L will always be larger or equal to DH_L. In the ideal case where $DH_L = CH_L$, each group in the lot is made of only one particle, or of several particles with identical critical content (Gy 1979). In the ideal case where CH_L is equal to zero, DH_L will consequently also be eliminated. DH_L can also theoretically be zero if the distribution of the groups of fragments in the lot is perfectly homogenous. However, there does not exist materials in science, technology or industry where this is the case. The primary role of a representative sampling process is to counteract DH_L, e.g. by employing composite sampling.

2.2.3 Sampling correctness

Sampling correctness is a fundamental requirement for allowing representative ore grade determination, effective process- and quality control, and valid feasibility studies (Pitard 1993). Only correct sampling processes can lead to representative samples, a prerequisite for reaching reliable conclusions from the analysis of extracted, composited and divided samples (Petersen et al. 2005). The Fundamental Sampling Principle (FSP) defined by TOS, states that all particles of the lot should have equal and non-zero probability of being extracted, in both the primary and all subsequent sampling stages. FSP stipulates that all virtual increments shall have an equal probability to be sampled. Furthermore, the integrity of the extracted increments/samples should always be fully respected, so that it is not contaminated in any way, nor affected by spillage, evaporation or other changes (Gy 2004).

Any sampling process will be associated with sampling errors (leading to sampling bias and/or uncertainties), meaning that the estimated grade of a sample will never be exactly equal to the true grade of the lot. The deviation of the sample grade (a_s) from the true grade (a_L) is defined as the relative sampling error (e), Equation 5.

$$e = \frac{a_s - a_L}{a_L} \tag{5}$$

A sampling process is deemed *accurate* if the average relative sampling error (m_e) is below a predetermined low threshold. For the sampling process to be rated as *reproducible*, or sufficiently precise, the variance of the relative error (s_e^2) should similarly be below a predetermined acceptance threshold. A sampling process can only be defined as *representative* when both these conditions are met, i.e. that the sample extraction is both accurate (the sampling bias is negligible or sufficiently small) and precise (the sampling variability is sufficiently low). The random part of the sampling error can often be reduced by for example increasing the sample mass, or by extracting a larger number of increments (composite sampling). However, the only way to ensure an accurate sampling process is to eliminate all bias-generating sampling errors, which can always be achieved by designing a TOS correct sampling process (although this may sometimes involve a considerable effort).

In practical sampling, the application of correct sampling processes is most easily achieved when extracting samples from 1D lots. Therefore, the primary objective when designing a sampling protocol is to identify a sampling location where the lot structure is one-dimensional, rather than for example three dimensional. Hence, it is preferable to sample the lot on a conveyor belt before it is transferred to a storage or stock pile, or in a pipeline before or after a slurry is transferred to a blender tank. In addition, the increment extraction should preferentially be realised at a transfer point, where the lot material is free falling and a complete cross section is easily accessible. Extracting complete cross sections of the lot to be sampled, at regular time or mass intervals, is a practical way of respecting the FSP.

2.2.4 Sampling errors

All sampling operations are error-generating processes (Gy 1979), subjected to nine types of sampling errors defined by TOS, Figure 12, (Esbensen and Paasch-Mortensen 2010, Pitard 2009). An error is defined as the difference between an observed/measured value and the true lot value, i.e. variations in measurements due to mistakes or uncontrollable factors. In contrast, uncertainty (the term most often used in statistics) is defined as lack of sureness, i.e. something that is not constant or include variation (Pitard 2009).

TOS describes both sampling errors leading to sampling bias if not eliminated, and sampling uncertainties leading to inflated variability if not minimized, but deliberately defines all of these as *sampling errors*. Even though all sampling errors are not literally mistakes, the word error has been retained for the sake of simplicity and to enforce the importance of handling all sampling errors to achieve representative sampling processes (Gy 1979, 1998).

Sampling errors are a consequence of the interaction between the sampling process and the heterogeneity of the material to be sampled. The sampling errors will, if not sufficiently eliminated and/or minimized, result in critically biased samples with unnecessarily inflated sampling variance. The Global Estimation Error (GEE), associated with a complete measurement system (including sampling, sub-sampling, preparation and analysis), is the sum of the Total Sampling Error (TSE) and the Total Analytical Error (TAE) respectively. Traditionally in mining and mineral processing, comprehensive monitoring and control of TAE has been the main focus, while comparable knowledge and competence about TSE is often limited. TSE is divided in two distinct groups defined by their specific nature, Figure 12.

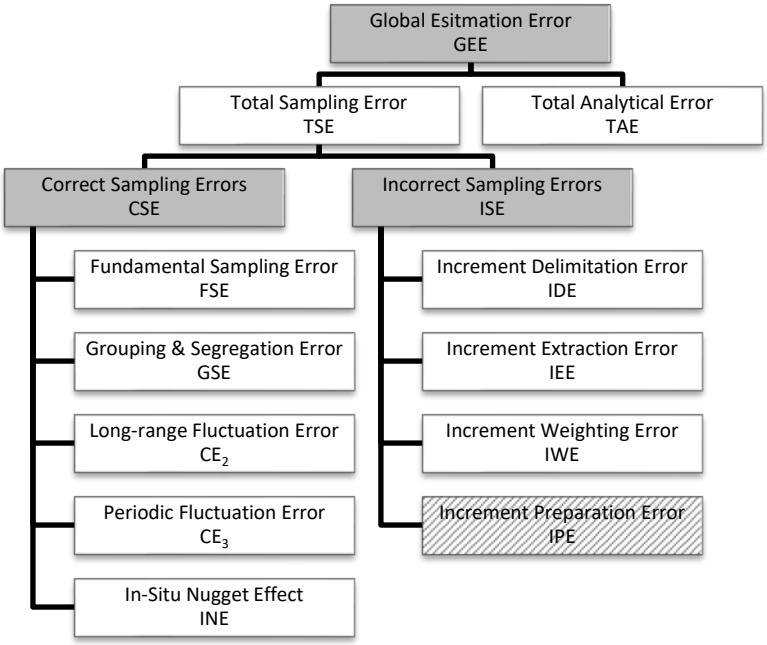

Figure 12. Classification of sampling errors according to their origin. Either stemming from the heterogeneity of the sampled material or from the materialisation of the sample increments as extracted by the sample equipment (derived from Gy 1979 and Pitard 2009).

The Incorrect Sampling Errors (ISE) are due to wrongful delineation, materialisation and/or preparation of the primary increments, leading to a critical sampling bias if not eliminated. As material heterogeneity is not a constant or uniform property, incorrect delineation, extraction and/or preparation will lead to adverse and unnecessary effects. Heterogeneity is inherently variable, depending on the physical and chemical properties of the material as well as the level of mixing or segregation. Thus, a bias stemming from an incorrect sampling process will also be variable and can therefore not be quantified or corrected for. In practice, this means that non-eliminated ISE leads to sampling biases that will inflate the total measurement system variability unnecessarily. This variability cannot be reduced by means of for example increasing the sample mass, number of increments, or replicating the analysis (Holmes 2010). However, ISE can be eliminated by applying TOS correct sampling processes and correctly designed, implemented and maintained sampling equipment. ISE can occur at all sampling stages and hence need to be eliminated throughout the entire sampling process to ensure representative samples and measurement results.

By way of contrast, the Correct Sampling Errors (CSE) are a manifestation of the heterogeneity of the sampled material. CSE can never be fully eliminated but should be minimized to allow sufficiently precise measurement results. CSE reflect how the increments are extracted from the lot, i.e. how the sampling equipment is designed and utilised, specifically to counteract the lot material heterogeneity. CSE are also present in all sampling stages following the primary sample extraction.

Non-eliminated ISE will lead to a sampling bias (non-accurate sampling), while CSE lead to an inflated sampling uncertainty (non-reproducible sampling) if not sufficiently minimised. Therefore, the only way to achieve representative, i.e. accurate and precise measurement results, is to apply TOS-correct sampling procedures that eliminate all ISE and sufficiently minimise all CSE, throughout the entire sampling process.

Correct Sampling Errors (CSE)

There are five CSE defined by TOS. The term *correct* error is sometimes considered to be contradictory, but the term simply refers to that these errors are the ones left after the *incorrect* sampling errors have been eliminated. The CSE need to be sufficiently minimized to achieve representative samples but will always contribute with some level of uncertainty to the final measurements (in addition to the analytical uncertainty). CSE consists of the Fundamental Sampling Error (FSE), the Grouping and Segregation Error (GSE), the two heterogeneity fluctuation errors related to long-range trend variations (CE_2) and periodic variations (CE_3) respectively, and the In-Situ Nugget Effect (INE), equation 6.

$$CSE = FSE + GSE + CE_2 + CE_3 + INE \tag{6}$$

FSE and GSE are present in all practical sampling situations for all lot dimensionalities. However, CE_2 and CE_3 are reflecting longitudinal heterogeneity, introduced by trends and periodic variations, producing some degree of autocorrelation between extracted increments, in 1D process sampling situations. In process sampling, the sum of FSE and GSE, i.e. the errors stemming from the CH_L and DH_L of the sampled material, is also termed the short-range heterogeneity fluctuation error (CE_1), Equation 7 (Pitard 1993).

$$CE_1 = FSE + GSE \tag{7}$$

FSE is directly related to the CH_L of the lot, whether it is stationary or moving. FSE alone is the minimum possible sampling error that can ever be achieved. FSE can only be fully eliminated by analysing all particles of the lot, which naturally is impossible in practical applications. Thus, FSE is the singular sampling error remaining, to some degree or other, even when all other sampling errors have been eliminated. Mixing, or so-called homogenisation, has no effect on CH_L and therefore not on FSE, which is dependent on factors such as mineral composition, liberation, shape and fragment size distribution of the lot (Pitard 1993). Pierre Gy has derived a practical link between FSE and observable material characteristics, that has become known as Gy's formula. This thesis will not further develop the mathematical concepts around FSE and Gy's formula. However, these concepts are comprehensively described in various TOS literature (e.g. Gy 1998, Pitard 1993).

GSE is related to DH_L that describe the variability between increments in the lot (Pitard 1993). GSE is dependent on the size of the extracted increments and their spatial disposition in the lot and can be reduced either by extracting a larger number of smaller increments and/or by effective mixing of the material in the lot before sampling. It is essential to avoid introduction of any additional errors, e.g. IDE or IEE, in the attempts to reduce GSE. In process sampling applications, there is often only little practical possibility to reduce the segregation factor of GSE through mixing (Gy 1979). The smallest possible GSE can only be

achieved under the ideal condition where fragments are collected one by one, selected at strictly random, which is practically impossible to achieve. Samples are instead normally made up by a number of extracted increments (composite sampling), each of which are made up by several fragments. Therefore, with TOS-correct procedures, samples are made up by randomly extracted increments, or groups of fragments.

The additional CSE, CE_2 and CE_3, are process sampling errors introduced by the long-range heterogeneity present in dynamic 1D lots. These will, together with CE_1 (defined as the sum of FSE and GSE in process sampling contexts), be further addressed in section 2.4. The In-Situ Nugget Effect (INE) is related to particles or clusters of the constituent of interest in unbroken and undisturbed material exposed to e.g. core drilling. INE is an error that is only recently defined by TOS (Pitard 2009). INE only impacts the sampling process before the material is broken up into smaller particles. However, it has large implications in mineral economics, for example during feasibility studies. (Note that INE is not equivalent to the nugget effect (*V(0)*) described by the variogram, see 2.5.2, but rather constitute a part of the components making up the variogram nugget effect).

Incorrect Sampling Errors (ISE)

The notion of *incorrect* sampling errors reflects the properties of these errors as being generated by incorrect sampling procedures. The incorrect sampling errors can only be fully eliminated if correct sampling procedures, following the rules described by TOS, are applied. This contrasts to the *correct* sampling errors, that can be minimized but not eliminated, even with TOS correct sample processes. The ISE are also called materialisation errors as they are introduced by the sampling equipment interacting with the material as increments are extracted from the lot.

The Increment Delimitation Error (IDE) is related to the design of the sampling equipment used to extract increments from the lot. A correct delimitation of a one-dimensional lot increment is a complete cross section of the material stream, easily accessed at a conveyor belt or pipeline transfer point and delineated by parallel planar or curve-planar boundaries, Figure 13. Correct delimitation is achieved when a sample cutter transgresses the lot with uniform speed and continues to have parallel edges also when intercepting the complete width-depth of the material, i.e. when all elements of the cross section are intercepted by the sampling cutter in the same length of time (Gy 1979). If the increment delimitation is incorrect, IDE and therefore a sampling bias will be introduced.

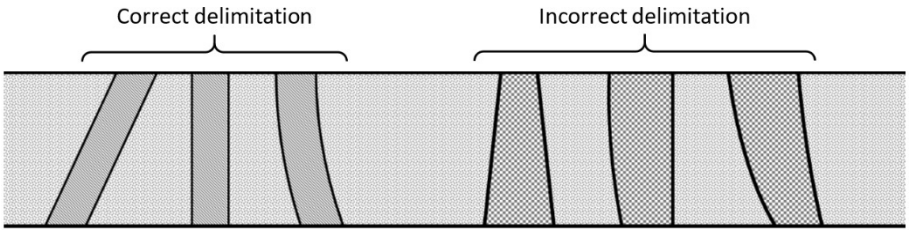

Figure 13. Examples of correct and incorrect increment delimitation from a 1D lot.

The Incorrect Extraction Error (IEE) refers to the physical extraction of increments. Even though the delimitation of an increment is correct, incorrect physical sampling operations can lead to incorrect sample extraction. The so-called *rebounding rule* and *rule of centre of gravity* need to be respected in order to eliminate the IEE (Gy 1979). The rebounding rule means that all particles delineated by the sample cutter should end up in the extracted increment, i.e. that the sample cutter edges and opening shall be designed so that no particles colliding with the edges or walls of the cutter bounce free off the cutter and exit the increment. The rule of centre of gravity refers to that all particles having centre of gravity within the delimited increment should also end up in the extracted increment, while particles with centre of gravity outside the delimited increment should be rejected by the sample cutter (Pitard 1993). Some practical aspects for reaching correct increment extraction include i) straightness, thickness, shape, length and inclination of cutter edges, ii) cutter width and speed and iii) cutter depth, capacity and design.

The Increment Weighting Error (IWE) is also related to how increments are extracted. IWE is introduced if the extraction of increments is non-proportional to the flow of material being sampled. Elimination of IWE is achieved either through i) mass-basis sampling, where each increment is of uniform mass and the increment spacing is determined as a fixed mass interval or ii) time-basis sampling, where each increment mass is proportional to the flow rate of the material stream and the increment spacing is determined as a fixed time interval (Pitard 2009).

The Increment Preparation Error (IPE) is not strictly speaking a sampling error, as it is not related to the selection process where increments are extracted from the lot, but rather occurs in-between sampling stages and/or after sampling is completed. However, as IPE (if present) will introduce a bias and is related to the practical implementation of the sampling protocol, it is often referred to as a sampling error together with the remaining three ISE (Pitard 2009). Activities that can introduce IPE are sample transfer spillage or contamination, unwanted comminution, drying or evaporation, accidental filtration or fractionation. The adverse effects of IPE, e.g. contamination, loss of fines and/or alteration of chemical and/or physical composition are essential to avoid and it is imperative to eliminate all IPE in order to ensure the sample integrity throughout the complete sampling process and hence achieve a representative analytical aliquot.

2.3 Open pit mine sampling

Open pit mining is used for ore deposits where the economically minable ore is located near the surface, in contrast to underground mining, used when the economically viable ore is found deeper below the surface. Open pit mining at LKAB is performed using benches of 15 metres thickness, where vertical holes are drilled, charged with explosives and blasted. The current mine grade control at LKAB is based on geological 3D and resource block models derived from diamond drilling, enhanced with Blast Hole drill sampling (BH sampling), Figure 14, and analysis for increased detail.

The most commonly used methods for open pit mine grade control around the world are BH sampling and Reverse Circulation drill sampling (RC sampling). Several publications have evaluated one or both sampling methods in the quest of establishing a general conclusion as to a best approach with respect to representativity (e.g. Abzalov et al. 2010, Chieregati et al. 2011, François-Bongarçon 2010, Gomes et al. 2011, Pitard 2008). These evaluations typically show varying and partly opposing results depending on the mining situation, ore composition and specific drill rigs and sampling equipment used. It is noticeable that several of the published evaluations start with the preconception that RC sampling is representative and therefore use this method as a reference with which to compare BH sampling (e.g. Caccioppoli and Candy 2011, Chieregati et al. 2015). However, other studies indicate that RC sampling is not necessarily a representative sampling method (e.g. Abzalov et al. 2010, François-Bongarçon 2010, Goers and Almond 2012). Another drill sampling approach is Diamond Drilling (DD), which is generally accepted as the superior sampling method for representative mine sampling and therefore often used for exploration purposes. However, as the drilling costs are significantly higher than for both BH- and RC sampling, DD is generally not used for production grade control in open pit mining.

Figure 14. BH drilling and sampling using a stationary radial bucket, during one of the variographic experiments conducted within this Ph.D. study. The pictured D65 drill rig utilises a cyclone to separate fines and dust to a separate BH cone in the back of the drill rig.

2.3.1 *Blast hole drill sampling*

Blast hole drilling generally uses a piston hammer and drill bit with pneumatic drive. The hammer is driven down in the ground with great force to fracture the rock and drill cuttings are flushed out of the hole with compressed air. During drilling, the drill cuttings are either discharged around the drill hole or gathered by a dust collector and passed through a cyclone (separating dust and fines to a separate BH cone) before being discharged close to the hole, Figure 14.

BH sampling most often includes manual extraction of samples from the cone of drill cuttings. Conventional methods for extracting BH samples include e.g. shovelling, spear sampling and collecting vertical slices from the centre of the cone or radial slices from the complete cone, all of which are subjected to some (smaller or larger) degree of ISE. In practical applications, a single BH sample per drill hole is normally collected. There are several problems related to sampling of BH drill cuttings, including the sub-drill (i.e. that the BH is drilled below the bottom of the bench to allow for correct fractioning during blasting), BH cone segregation, loss of fines and poor material recovery from the drilled blast hole, Figure 15 (Pitard 2008).

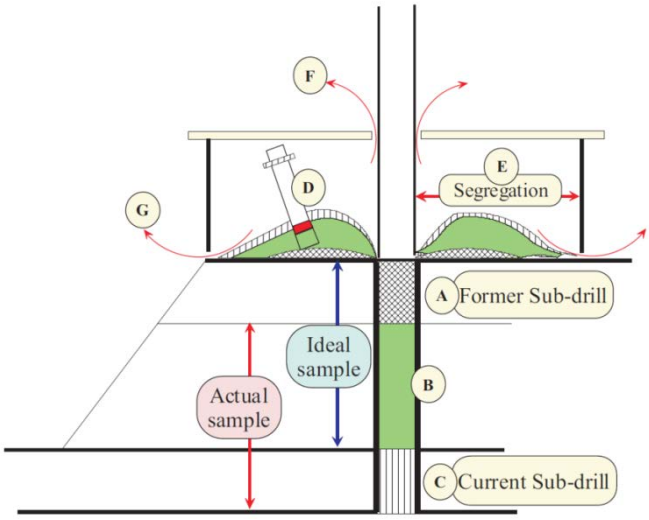

Figure 15. Summary of BH sampling problems, including problems with the sub-drill (A, C), BH cone segregation (D, E) and loss of fines (F, G) (reprinted with permission: Pitard 2008).

Some suggestions on how to reduce the sampling errors connected to BH sampling have been presented and some studies indicate that BH sampling can be deemed fit-for-purpose even though residual sampling errors are likely present (e.g. François-Bongarçon 2010, Gomes et al. 2011, Séguret 2015). The general solution to achieve fit-for-purpose samples from a BH cone of drill cuttings is to extract one or multiple radial increments. Methods for extracting radial increments include sectorial sample frames, spear samplers applied in a radial pattern or radial shovelling, Figure 16. These methods are designed to manage the problems with pile segregation, but none of them can handle the problem with the sub-drill, loss of fines during drilling or inadequate material recovery from the blast hole (Pitard 2008).

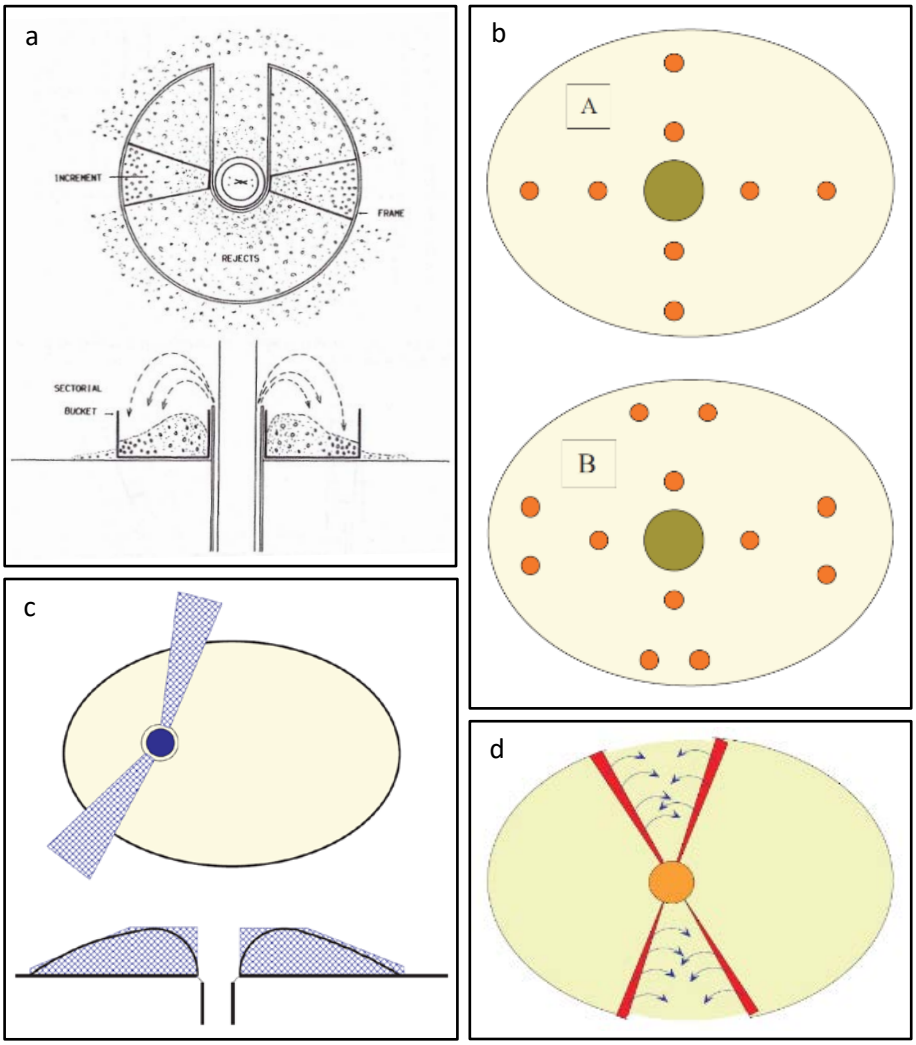

Figure 16. Some suggested methods for handling the problems related to BH sampling. a) Stationary sectorial sample frame/bucket, b) A: Spear sampling introduction IDE, B: Spear sampling simulating a correct radial cut, c) Correct design and positioning of radial buckets and d) Collecting four thin radial increments for a composite sample (reprinted with permission: a) Pitard 1993, b, c and d) Pitard 2008).

2.3.2 *Reverse Circulation drill sampling*

In contrast to BH drilling, RC drilling uses a rotary drilling technique with dual wall drill rods. The hollow inner tubes of the drill rods allow drill cuttings to be transported to the surface in a steady flow. The general method for extracting samples during RC drilling is to use a rotary divider or riffle splitter to reduce the drill cuttings to a desired sample mass. The RC sampling process generally results in several samples per drilled hole, for example one sample per drilled meter. This can be a desired feature for allowing detailed grade control, but also leads to a large number of samples and large sample masses to be handled by sampling and laboratory staff.

RC sampling has been found to be more representative than BH sampling in some studies (e.g. Caccioppoli and Candy 2011, Magri and Ortiz 2000, Pitard 2008). However, several problems related to RC drill sampling can lead to biased grade estimations, e.g. down-hole contamination, poor material recovery, increased drill-spacing and the plucking effect, Figure 17 (Abzalov 2010, Pitard 2004).

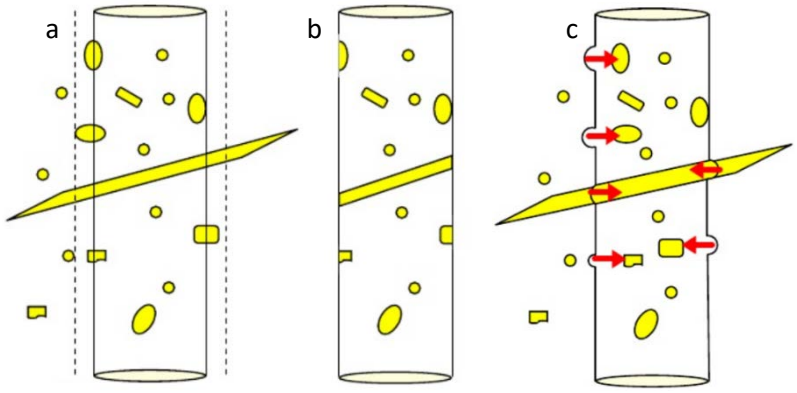

Figure 17. Plucking effect generated by RC and BH drilling. a) in-situ ore, b) ideal recovery and c) plucking effect (reprinted with permission: Pitard 2004).

2.3.3 *Alternative measurement solutions*

Alternative solutions for traditional grade control sampling and measurement systems have recently been developed for open pit mining application. One example is an in-situ measurement system using the Pulsed Fast and Thermal Neutron Activation (PFTNA) technique. PFTNA rely on a pulsed neutron generator that sends neutrons toward the walls of the drill hole. The neutrons penetrate the surrounding material, exciting gamma photons that are detected and translated to grades for the chemical constituents of interest for most mining industry applications (Smith et al. 2015). The PFTNA utilise a down the hole tool that lowers the measurement system in to the hole, for real-time in-situ measurement of the chemical grades of the material in the walls of the drill hole. PFTNA is critically dependent upon correct calibration and need to be adapted and tested for each individual application.

2.4 Process (1D) sampling

Process sampling is the most common practical application of sampling of 1D lots. Generally, process sampling refers to sampling from moving streams of material, e.g. particulate material on conveyor belts, slurries in pipelines or chutes, or sampling of discrete chronological units. The main characteristic of the 1D lot subjected to process sampling is that the lot is restricted in two dimensions (width and depth), while it is elongated and often vastly extended in the third dimension (Gy 1979). This allows for TOS correct increments to be extracted as complete cross sections of the material stream at regular intervals, to be combined to a composite sample, Figure 18. The 1D lots produced and sampled in process industries are normally affected by some degree of autocorrelation, meaning that extracted increments are more alike if the spacing between them is small. Hence, the autocorrelation is strong for closely spaced increments, while it is declining until non-existent as the increment spacing increase.

1D process sampling is, apart from the already described CSE and ISE, also affected by the heterogeneity fluctuation errors CE_2 and CE_3. These are related to variabilities originating from either human activities in the mine or processing plants, e.g. loading of heterogeneous material onto a conveyor belt, or to large-scale composition or chemical content trends or cycles in the ore body and processed material. This leads to variations in the process stream that not only stems from the material in itself (described by CE_1) but also from how the process is controlled (Minnitt and Esbensen 2017, Pitard 1993).

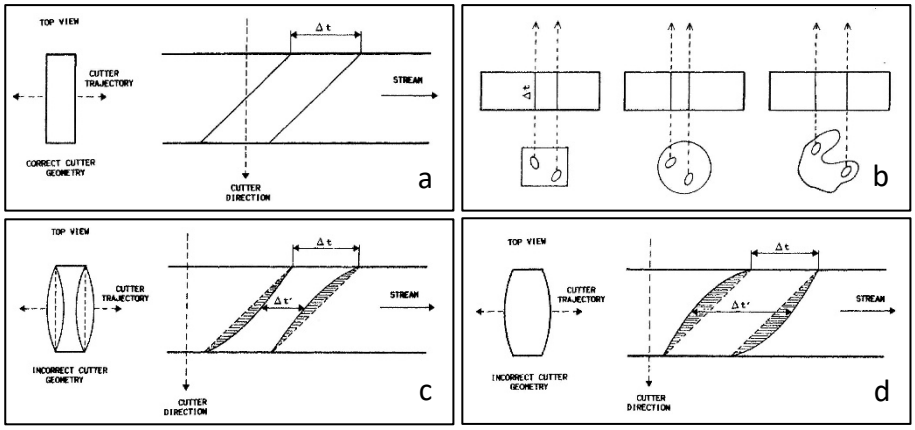

Figure 18. Examples of practical solutions for linear sampling of particulate material streams: a) correctly designed cross stream sampler that b) can handle any shape of material stream, c) cross stream sampler with built up material leading to IEE, d) cross stream sampler made of weak material, leading to non-parallel edges and IEE over time (reprinted with permission: Pitard 1993).

Practical process sampling implies extraction of several increments from a moving stream of material. The TOS-correct method of extracting increments is to collect a complete and uniform cross section of the material stream. The preferable way of achieving this is to transect the stream at a transfer point between conveyor belts or slurry pipelines, where the material is free-falling. To collect a complete cross section of a material stream by sampling from the top of a belt, using manual sampling methods or mechanical cross belt (hammer) samplers is not recommended as it is impossible to achieve a complete cross-section increment in this manner (ISO 3082 2009). There are almost countless numbers of practical and mechanical solutions for extracting increments from particulate material process streams, Figure 18 and 19, and slurry streams, Figure 20. However, several sampling systems available and applied today, do not conform to the principles of TOS-correct increment extraction. These incorrectly designed systems can be expected to result in an excessive sampling variability or a sampling bias.

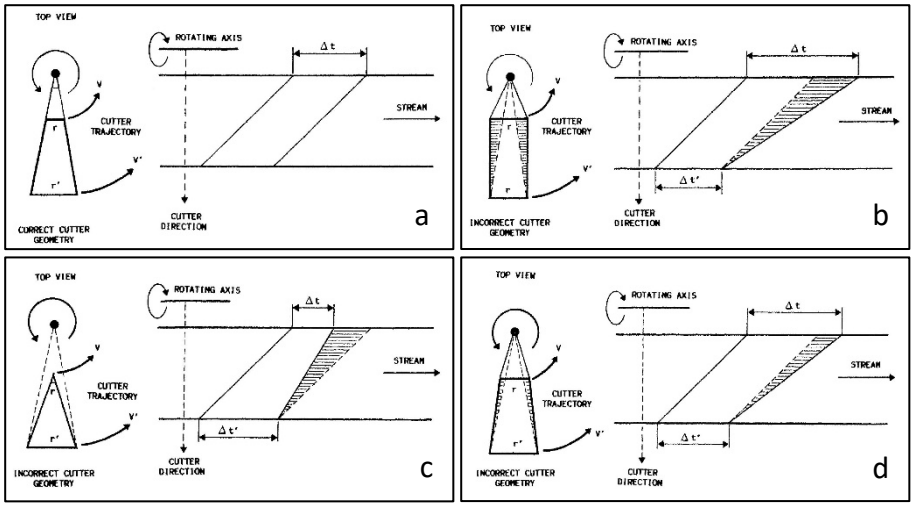

Figure 19. Examples of practical solutions for radial sampling of particulate material streams: a) correctly designed Vezin sampler with radial cutter edges, b) incorrectly designed Vezin-sampler with parallel cutter edges, c) incorrectly designed Vezin-sampler with non-radial cutter edges, d) incorrectly designed Vezin-sampler with non-radial cutter edges (reprinted with permission: Pitard 1993).

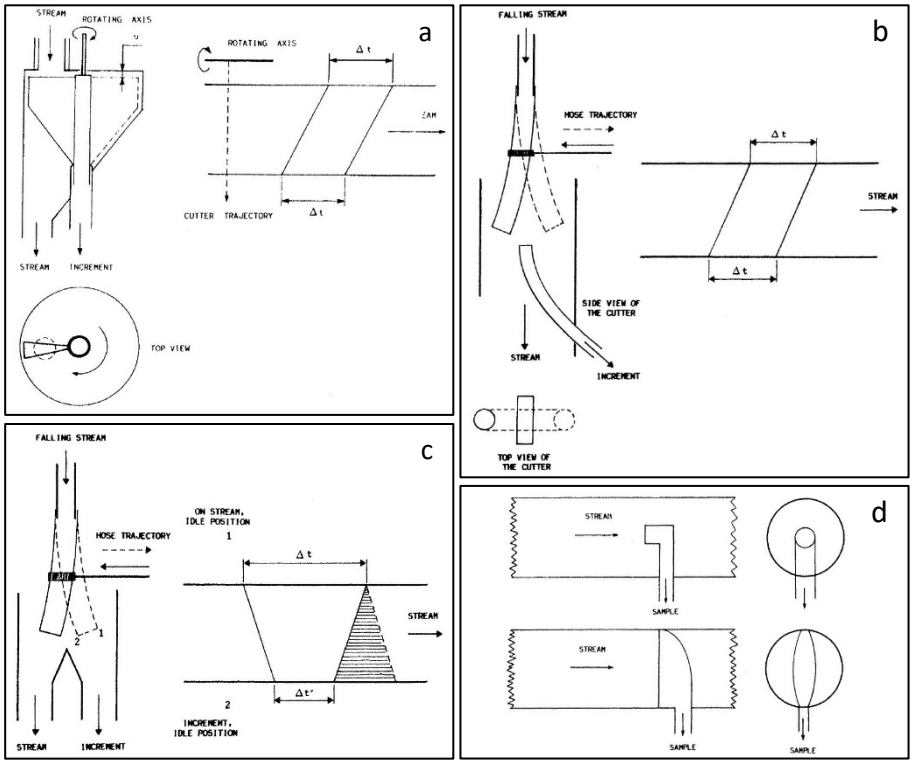

Figure 20. Examples of practical solutions for process slurry sampling: a) correctly designed Vezin sampler collecting a uniform cross section of the stream, b) correctly designed hose sampler collecting a uniform cross section of the stream, c) incorrectly designed hose sampler collecting a non-uniform cross section of the stream, d) incorrectly designed spear and shark-fin samplers only collecting part of the stream (reprinted with permission: Pitard 1993).

2.5 Methods for evaluation of industrial measurement systems

There are several statistical and experiential methods for assessing the performance of industrial measurement systems. Variographic characterisation, replication experiments and duplicate sampling experiments are three examples of well-established methods presented in numerous TOS related publications (e.g. Esbensen and Wagner 2016, Gy 1979, Lyn et al. 2007, Minnitt and Pitard 2008, Pitard 1993, Ramsey 1992). All three methods have been applied within this PhD study, to various measurement systems at LKAB iron ore operations. The choice of evaluation method is based on several parameters, e.g. scope of evaluation, time frame, data availability, sampling target, lot dimensionality and sample extraction method.

2.5.1 Bias testing

Bias testing is an important aspect for industrial sampling, as several international standards, e.g. for iron ore (ISO 3082 2009, ISO 3086 2006), advocate this method to check the accuracy of a newly installed or modified sampling system. This means that bias testing of sampling systems often is necessary in seller-customer relationships, in order to validate that the sampling and analysis are representative of e.g. a shipload of delivered products. However, bias testing, where an applied sampling system is compared to a reference sampling method (e.g. stopped belt sampling or full cone blast hole sampling), is always only a snapshot of a potential sampling bias. If a sampling bias is detected, this only gives information regarding the level of the bias at that specific instance. As all sampling biases are inconstant, a bias test can only determine if there is a sampling bias present or not but can never quantify the bias or enable any 'bias correction' as this is a structural impossibility.

The only certain method to ensure that a sampling system is representative and does not produce biased samples, is to apply TOS correct sampling processes. If the sampling method complies with all the pertinent TOS rules, the sampling system will be representative and the need for costly bias testing is eliminated. However, industrial sales contracts often require adherence to the applicable ISO standards, hence demanding comprehensive bias testing of customer sampling systems, as in the LKAB harbours. This requirement is already fulfilled at LKAB and bias testing of process or delivery sampling system is therefore not included in this PhD project. Furthermore, sampling bias manifestations will always be varying due to the inherent nature of lot material heterogeneity. This means that a sampling bias will lead to an inflated sampling variance or sampling uncertainty. Powerful methods for quantification of the total sampling and measurement variability are defined by TOS and presented below.

For open pit mine sampling, bias testing has been applied within this study, to compare various drill sampling methods to full BH cone reference samples. The purpose of these tests was to clarify how non-correct measurement systems can lead to sampling bias, as well as to compare this bias between various sampling methods. Due to the large amount of resources and time needed for bias testing, it is not possible to conduct such regularly, nor is it necessary when the total measurement system is not changed. However, the presented methods for evaluation of total measurement system variability are versatile for continuous evaluation of industrial sampling systems.

2.5.2 *Variographic characterisation*

Process samples are influenced by variabilities stemming from the material being processed, from the process itself and from the measurement system applied to describe the characteristics of the process or product. For complete understanding of the total observable process variability, it is important to be able to *discriminate* between the true process variability and the variability (uncertainty) stemming from the measurement system. There can be large variations in the ratio between these two variabilities for various process data series in technology and industry.

Variographic characterisation is a powerful tool, providing a structured way of quantifying the magnitude and origin of different variance components in process data. The semi-variogram enable determination of the so-called nugget effect (*v(0)*), which is an estimation of the variability stemming from the measurement system (including primary sampling, sub-sampling, sample preparation and analysis). Variographic characterisation can be applied to any 1D process data that is ordered in time or space, e.g. process monitoring data or similar experimental data, but also to space correlated drill sampling data (Esbensen and Paasch-Mortensen 2010).

The variogram can be calculated based on absolute analytical concentrations (a_q) or based on heterogeneity contributions (h_q), where the latter is termed the *relative semi-variogram.* The advantage of using heterogeneity contributions is that the relative variogram enable direct comparison between variograms representing various measurement systems and diverse analytical parameters. The basis for the term *semi*-variogram is the division by a factor 2, in order to express the variability as a standard statistical variance (Pitard 1993). Calculation of the heterogeneity contributions and point calculation (*v(j)*) of the relative semi-variogram follow equation 8 and 9, where *v(j)* is the variogram point estimations (expressed as statistical variances), h_q is the heterogeneity contribution for increment q, a_q is the analytical concentration for increment q, a_L is the mean analytical concentration of the lot, M_s is the sample (or increment) mass, Q is the total number of measurements and j is the lag (distance between samples or increments in time or space)

$$h_q = \left(\frac{a_q - a_L}{a_L}\right)\frac{M_s}{\overline{M_s}}. \tag{8}$$

$$v(j) = \frac{1}{2(Q-j)}\sum_{q=1}^{Q-j}\left(h_{q+j} - h_q\right)^2, \qquad j = 1,2,\ldots.,\frac{Q}{2}. \tag{9}$$

The variogram point estimations, *v(j)*, is calculated for each analytical spacing, (j = 1,2,3, …), Figure 21. The spacing is termed *lag (j)* and denotes the distance in time or space between two analytical results, i.e. for lag one (*v(1)*) the variogram is calculated for all analysis pairs with spacing j = 1. The variogram is calculated for all lags up to a maximum of j = Q/2, where Q is the total number of chronological data points available for the variogram calculation. This is to ensure that all analytical results in the data set is included in the variogram calculations. When j > Q/2, the central values of the chronological data set will not be involved (Pitard 1993).

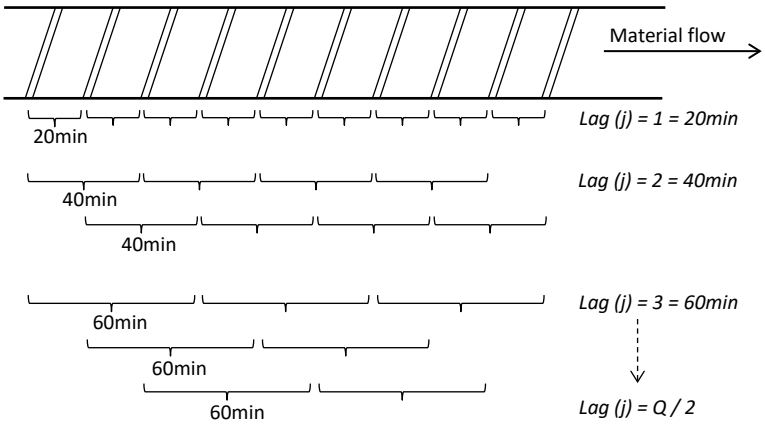

Figure 21. Schematic view of the basis for computation of a variogram.

Variograms can have distinctly different appearances, but three main parameters can be estimated and used for comparison between relative semi-variograms. These features are the *nugget effect (V(0)),* the *sill* and the *range,* Table 1 and Figure 22. In the 1D process variogram, the nugget effect is the sum of all variability contributions from the total measurement system (including primary sampling, sub-sampling, preparation and analysis). In practice this means that the nugget effect is an estimate of FSE, GSE, INE as well as all non-eliminated and variable ISE. Therefore, it is an important parameter for the evaluation of measurement system performance. The sill describes the total variability present in the variogram data set and the range describes the total lag distance to where the sill is reached.

Another important characteristic of the semi-variogram is the *nugget-to-sill* ratio, Table 1. This ratio describes how large the variability contribution from the measurement system alone (nugget effect) is in relation to the observable overall variability, reflected by the sill level (DS 3077 2013). The nugget-to-sill ratio is a useful quality index and a grading tool of the measurement system performance. The smaller nugget-to-sill ratio, often expressed as a percentage, the better performing is the particular process measurement system. The relative semi-variogram, with estimation of nugget-to-sill ratio, is therefore a powerful tool for assessing and comparing different process measurement systems. The full mathematical description for variographic analysis is presented in detail both in the historical TOS literature and in more recent publications (e.g. Esbensen and Paasch-Mortensen 2010, Gy 1979, Minnitt and Esbensen 2017, Pitard 1993). Practical application of variographic characterisation is applied in appended papers number II, III, V, VI and VII.

Table 1. Important parameters for characterisation of the relative variogram (Gy 1998, Minnitt and Esbensen 2017, Minnitt and Pitard 2008).

Notation	Definition	Description
***v(0)* - Nugget effect**	Extrapolated intersect of y-axis	Short range random variability stemming from the total measurement system, including sampling, sub-sampling, sample preparation and analysis. The nugget effect is the summation of material heterogeneity, FSE and GSE, as well as all non-eliminated ISE stemming from incorrect sampling procedures. The nugget effect is approximated by extrapolation of the variogram to the y-axis, i.e. the fictive point of *v(0)*.
Sill	Total variability in the data set	Describes the total variability, or the global heterogeneity, in the data set. Technically the sill is the average of all variogram point values. In an increasing variogram, the sill is similar to the level where the variogram flattens out.
Range	Where the variogram reaches the sill	For process variograms, the range can be directly translated into a time interval where paired samples show autocorrelation. The range is the point where an increasing variogram is flattening out, i.e. the lag distance where the variogram reaches the sill.
Nugget-to-sill ratio	Relative measurement system variability	The nugget effect divided by the sill describe the variability stemming from the measurement system relative to the overall variability. This ratio is an effective quality grading for any measurement system, also allowing distinct comparison between measurement systems.
v(1)	Value of *v(1)*	The non-random variability occurring in the process during the time period between two analyses, i.e. the sum of the total measurement system variability and the process variability that occurs between any two consecutive analyses.
v(Process)	*v(1) – v(0)*	The 'true' process variability for the time interval between two samples, not including the total measurement system variability. This is simply the difference between *v(1)* and *v(0)*.

The variogram appearance varies depending on the inherent process data characteristics, i.e. depending on the measurement system, the material heterogeneity and the process variabilities present during the time the variogram data were obtained. For positively autocorrelated process data, the variogram (*v(j)*) is expected to increase as the spacing between measurements increases, i.e. when the lag (*j*) increases, Figure 22. For data series with no autocorrelation (i.e. statistically independent data), the variogram appearance is flat. This may be due either to a high measurement system variability, to low process variability, or due to lack of closely spaced data, Figure 22 (Pitard 1993). The variogram can also exhibit a periodicity, stemming from a cyclic variation in the process data. The variogram will clearly show also small and diffuse periodicities, which are often hard to discern in the original process data, Figure 22.

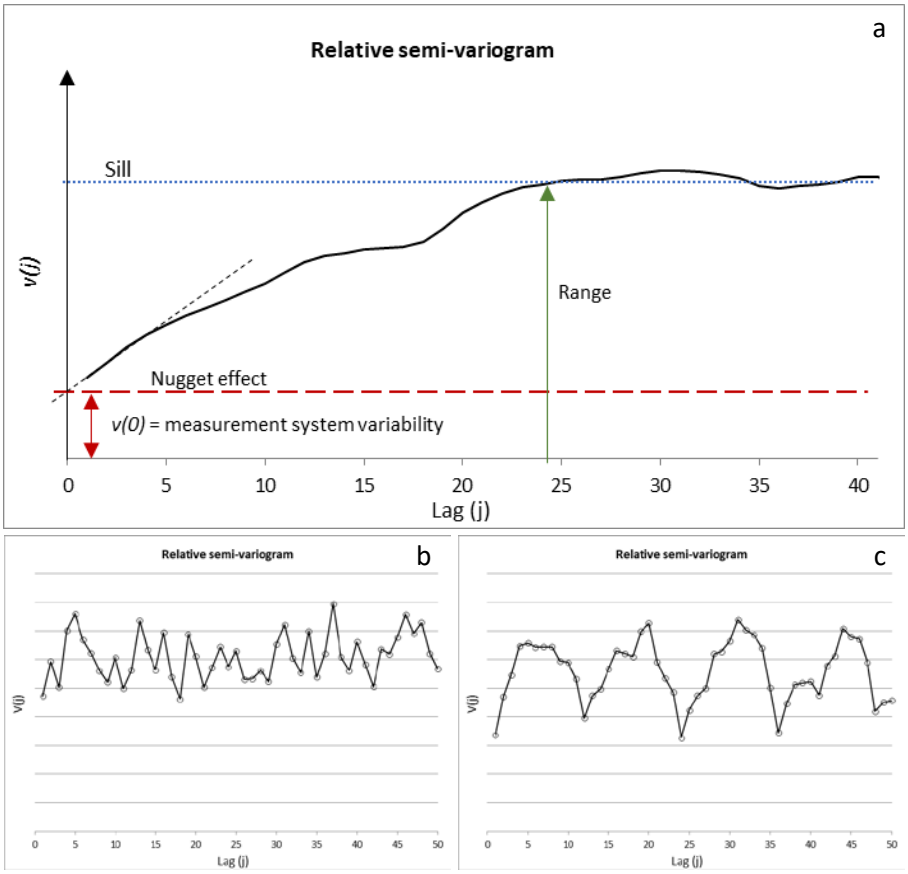

Figure 22. General appearances of various semi-variograms, a) general appearance of an increasing variogram, with visualisation of the three characteristic features: nugget effect, sill and range, b) flat variogram, c) periodic variogram.

2.5.3 Replication experiment

Replication of measurements has a long tradition for estimating uncertainty in the realm of chemical laboratories and design of experiments. However, even though analysis replication will give useful information of the analytical repeatability or decrease the analytical uncertainty if used for averaging, it will not reveal any information about the total measurement system performance as concerns primary sampling, sub-sampling or sample preparation.

A complete measurement system includes primary sampling, secondary sampling (sub-sampling/mass reduction/sample division) and sample preparation, before reaching an analytical aliquot ready for insertion in the analytical instrument, Figure 23. As primary sampling, secondary sampling and sample preparation often introduce significantly larger variabilities than the analysis itself, it is critically necessary to include these activities in a comprehensive *replication experiment,* to enable full evaluation of all uncertainties affecting the total measurement system (DS3077 2013, Esbensen and Wagner 2016). Furthermore, to allow for a complete decomposition of variability stemming from different sampling stages, replication can be deployed at several stages, i.e. replication of both primary sampling, sample preparation and analysis. Replication of the primary sampling step will allow estimation of the complete sampling error including both CSE and ISE, i.e. sampling uncertainties and non-eliminated sampling bias.

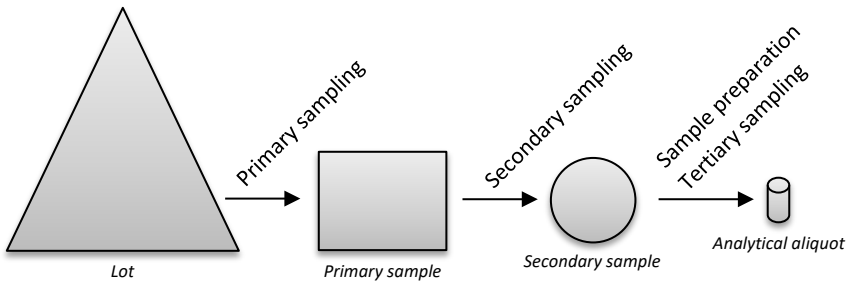

Figure 23. Measurement system stages before reaching the analytical aliquot. Replication can be performed in any one stage, but preferably should be done "from the top", i.e. from the primary sample extraction, in order to reflect the global estimation error including all sampling errors and the analytical error.

Replication experiments are especially useful when variographic characterisation is not applicable due to the lot dimensionality, insufficient increment lag spacing or a lack of data availability. The replication experiment is readily applicable to any sampling situation, not only for process measurement systems, with a minimum of additional effort. When replicating from the primary sample extraction, the aim is that the replicates should cover the entire spatial geometry and/or temporal variability as best possible (in order to guarantee compliance with the FSP), to allow the estimation of all possible variability that the sampling protocol may introduce (Esbensen and Wagner 2016).

For evaluation of the sampling replication experiment results, the relative coefficient of variance (CV_{rel}), also termed the *Relative Sampling Variability* (RSV), is used, equation 10. Where σ is the standard deviation and $\bar{X}$ is the mean of the measurement results from the replication experiment. The estimated *RSV* includes all non-eliminated sampling and analytical errors as manifested by the sampling process being assessed.

$$CV_{rel} = 100 \cdot \left(\frac{\sigma}{\bar{X}}\right) = RSV \qquad (10)$$

2.5.4 Duplicate sampling experiment

The duplicate sampling method is another straight forward method for evaluation of the variability stemming from primary sampling, sub-sampling, sample preparation and measurement, in relation to the overall variability in the data (Ramsey et al. 1992). To allow for all possible variability to be included in the evaluation, all duplicate sample sets should be collected in strict accordance to the sampling protocol under evaluation – identical to the primary sampling replication experiment stipulations. This means that all ambiguity in the sampling protocol should be accounted for in the physical and/or temporal spacing between extracted samples. To enable full discrimination of the variabilities stemming from primary sampling, sample preparation and analysis, the duplication should be repeated at all sample preparation and/or analytical stages, Figure 24 (Lyn et al. 2007). The Robust Analysis of Variance (RANOVA) is recommended for evaluation of the duplicate experiment. In contrast to classic Analysis of Variance (ANOVA), RANOVA is not as sensitive to small numbers of outlying values due to sampling, geochemical or technical issues (Ramsey et al. 1992).

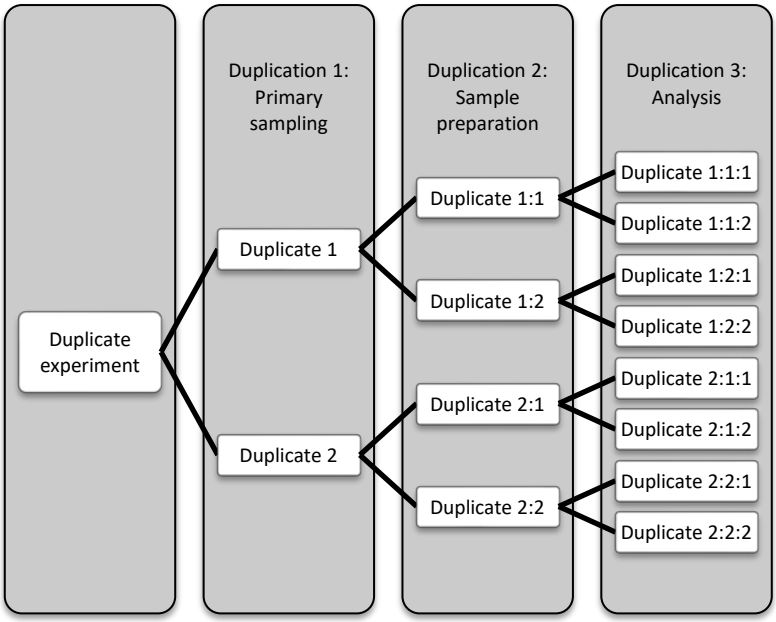

Figure 24. Visualisation of a hierarchical duplicate sampling experiment with duplication of primary sampling, sample preparation and analysis.

2.6 Chemometrics and PCA

Raw data from industrial processes and experiments cannot always be used for direct interpretation until appropriate data analysis is applied. Therefore, proper definition of the analytical problem is essential. The possibility to extract useful information from the available data depends on the actual content of assumed information, relative to the purpose of the measurements. A fundamental basis for optimising the possibility of proper conclusions, is to measure meaningful variables rather than to simply perform a large number of measurements (Esbensen 2009, Esbensen and Swarbrick 2018).

Empirical sciences and process industries, including mining and mineral processing, often generate multivariate data that need to be evaluated with appropriate methods to enable valid conclusions. A traditional and powerful method for describing multivariate data is Principal Component Analysis (PCA). PCA uses orthogonal transformation of the original data set, into a set of uncorrelated linear combinations of the original variables. These orthogonal transformations are expressed in terms of principal components (PCs). The first few PCs describe the largest proportion of variance in the original data. This allows the underlying data structure to be modelled with fewer of these *latent* variables, allowing important information to be assessed and evaluated based on a radically lower set of dimensions, while at the same time effectively disregarding random noise. PCA is widely used for finding hidden data structures, discriminate between systematic information and random noise, as well as detecting outliers and visualise patterns and correlation between variables and samples (Esbensen and Geladi 2009, Esbensen and Swarbrick 2018).

There are four elements in a PCA model: raw data, scores, loadings and residuals. These are preferably visualised through plots, which provide possibilities for both pattern detection and discrimination between objects and variables. The score plots present optimal possibility for detecting groupings, trends and irregular patterns among the samples in the original data. The loading plots show which variables affect the calculated components as well as variables that do not add to the specific variability along the same component(s) (Bro and Smilde 2014, Esbensen and Swarbrick 2018). The complement of a loading plot enables clear visualisation of the groupings observed in the score plot, Figure 25.

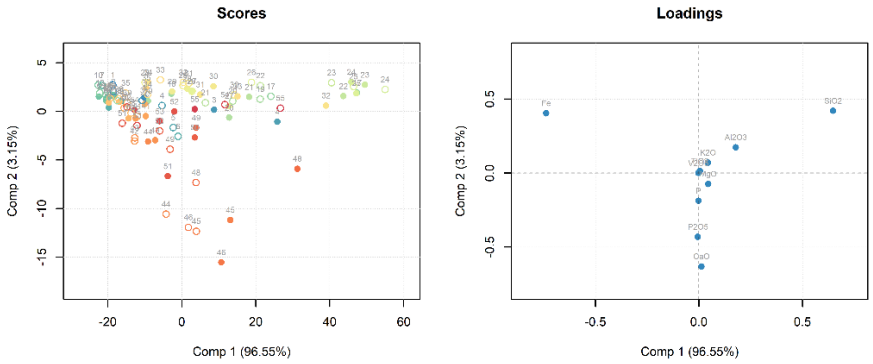

Figure 25. Visualisation of scores and loadings for an example PCA of chemical analysis data.

3. Papers prelude and conclusions

This chapter presents a summary of the appended papers, including important results and conclusions. The appended papers are based on the theoretical background compiled in chapter 2 and utilise the presented empirical methods to enable comprehensive assessment of the performance of both current and alternative sampling and measurement systems within LKAB iron ore operations. Furthermore, overall conclusions drawn from this PhD project, as well as recommendations for further work, are presented in this chapter.

3.1 Papers prelude

This study spans a wide range of vastly different sampling processes and measurement systems applied to various sampling targets at the LKAB iron ore operations in northern Sweden. From grade control in the open pit mines, throughout the full processing value chain, to the environmental surface water sampling of recipient water streams. The common denominator for all these applications is that representative measurement results are the critical success factor for reaching reliable conclusions and enabling valid decision making.

The seven appended papers in this thesis focus on evaluation and optimisation of sampling systems, to conclude if they are fit-for-purpose or need improvement. Apart from the literature study in paper I, which relies purely on previously published results, all papers include empirical case studies and experiments, using existing process data and/or experimental data from measurement systems employed at LKAB. Variographic characterisation has been the general approach for measurement system evaluation, while two studies, paper II and IV, have been supplemented with replication and duplicate sampling experiments.

While the focus of each individual paper has been to evaluate and optimise the selected sampling processes studied in that specific case, one general objective of this PhD has been to evaluate the applicability of variographic characterisation in mining- and mineral processing operations. Based on the comprehensive results from the presented case studies, the general possibilities and benefits of variography are discussed in the concluding sections of this thesis.

3.2 Summary of results

3.2.1 Open pit mine sampling (papers I, II and III)

Paper I, II and III of this thesis concern the performance of various methods for open pit mine drill sampling. The three papers are presented as a sequence of increased understanding of available open pit mine drill sampling methods and how they perform in regard to sampling bias and sampling variability in general, and more specifically in the iron ore open pit mining at LKAB operations in northern Sweden.

Paper I: A comprehensive literature review reflecting fifteen years of debate regarding the representativity of reverse circulation vs blast hole drill sampling

The study of open pit mine sampling was initiated with a comprehensive literature review reflecting fifteen years of debate regarding the representativity of Reverse Circulation drill sampling (RC sampling) and Blast Hole drill sampling (BH sampling). The literature review located a total of 31 publications from the years 2000 – 2017. 16 papers were published in conference proceedings from specific sampling conferences, while 10 papers were from other mining conferences and five papers were published in peer-reviewed journals.

Most of the reviewed papers evaluated BH sampling or compared BH and RC sampling, while four of them only evaluated RC sampling. The literature review showed that the performance of BH- and RC sampling is strongly related to the ore deposit and mining conditions at hand. Several problems with BH sampling, e.g. loss of fines, upward/downward contamination, pile segregation, pile shape irregularities, operator dependent sampling and non-probabilistic sampling equipment, were presented. Methods for reducing some of the problems exist and were also discussed in several publications. Sampling grid resolution was, in several publications, concluded to be more important than sampling performance. The increased sampling grids can lead to more misclassified mining blocks with RC sampling, in contrast to BH sampling, which often utilise more detailed grids. While some studies concluded that BH sampling can be fit-for-purpose, if care is taken to develop satisfactory sampling procedures, others concluded that RC sampling is superior and enable increased profits. Some publications concluded that the increased cost of RC-drilling is motivated and leads to increased profit, especially in precious metal mining, while others showed that both RC and BH sampling is afflicted by sampling errors leading to non-representative samples.

The results in the publications varied considerably and the literature review infer that it is not possible to reach an overall conclusion about one drill sampling method being superior to the other. Both BH- and RC sampling have advantages and disadvantages that need to be assessed in connection with the ore type and mining conditions at hand. Therefore, it is necessary to evaluate the applied drill sampling method locally to ensure that no hidden economic losses are generated due to non-representative samples.

Paper II: Blast hole sampling (replicate and variographic experiments) in LKAB open pit iron ore mines: fit-for-purpose representativity?

The second paper of the open pit mine drill sampling study presents an empirical evaluation of the current manual BH sampling method applied in the LKAB operated open pit mines. The study includes one replication experiment and two variographic experiments. The objective was to evaluate the variability of the current manual sampling procedure applied to the blast hole cones after drilling is finalised, as well as an alternative radial segment sampling method. The study also furthered comparison with full cone BH reference samples to evaluate the presence of any sampling bias. The drill rig used in the study was an Atlas Copco D65 rig usually employed by LKAB. The D65 uses a dust collector that separates the coarse and fine drill cuttings in to two separate BH cones, Figure 14.

The variographic experiments in this study were performed in the Leveäniemi open pit mine. Canvases were placed under the two drill rig discharge chutes and a stationary sectorial cutter was placed under the coarse discharge. After drilling of each blast hole was finalised, a manual BH sample was extracted using the current sampling procedure and a radial segment sample was collected from the sectorial cutter. From 18 of the 55 blast holes, the complete fines and complete coarse BH cones were also collected, weighed and split, to enable calculation of a reference measurement result (weighted average) based on the four partial samples.

The results indicate that the manual BH sampling procedure show smaller deviations from the reference sample, for iron, silica and vanadium grade, compared to the segment sampling method. A significant problem with both the manual BH and segment sampling method is that they only collect material from the coarse BH cone. As the fines and coarse cone exhibited large deviations in chemical grade this leads to a sampling bias affecting both sampling methods. The variographic results indicate that the nugget effect, for iron and vanadium grade, for both the manual and segment BH sampling methods correspond to ≈ 20% of the total variability visible in the sill. This indicates that both sampling methods can produce sufficiently precise measurement results. The results from the replication experiment of the manual BH sampling showed that the Relative Sampling Variability (RSV) was below eight percent for the key variables iron and vanadium grade for all 24 completed replication experiments. The general trend was that the RSV was lower for areas in the mines with higher iron grades, indicating that the ore heterogeneity in these areas is lower, generating lower sampling variability.

This study concluded that the manual BH sampling method employed by LKAB performs well regarding sampling variability, i.e. the method is reproducible. The manual sampling method also shows better agreement with the reference measurement results than the segment sampling method does. However, for blast holes with low iron grade and therefore higher ore heterogeneity, the deviation from the reference result and the RSV is significantly larger than for blast holes with high iron grade. This is likely due to the manual sampling method excluding the fines BH cone in sample extraction. Hence, a sampling method applied before the dust collector, that separate the fines and coarse drill cuttings, on the D65 drill rig would be of distinct interest to improve the representativity of the BH sampling.

Paper III: Optimal grade control sampling practice in open pit mining – A full-scale Blast Hole vs Reverse Circulation variographic experiment

The third paper related to open pit mine sampling was a direct follow up study of the second paper. This study included a parallel variographic characterisation, employing RC drill sampling as twin holes to the BH sampling variographic experiment presented in paper II. This study applied Principal Component Analysis (PCA) to allow multivariate comparison between all chemical grades from the BH- and RC sampling respectively.

The results from the PCA indicated that the major variability in the data was described by PC1, that account for approximately 56% of the total data set variance. PC1 was strongly correlated to the Fe/V vs Si variability that is significant for the Leveäniemi ores. PC2 added 30% to the modelled variance, driven by phosphorus and calcium. PC3 described an additional 8.5% of variance, solely due to titanium variabilities. The PCA was conducted to compare the data from the BH and RC sampling methods and showed a smaller deviation between the sampling methods for drill holes with high iron grade than for drill holes with higher titanium, silica, phosphorus and calcium grades. A univariate comparison for iron and silica grade supports these results and showed larger discrepancies between the sampling methods as well as compared to the full cone reference results, for drill holes with lower iron grade and higher concentrations of apatite, calcite, actinolite, ilmenite and titanite, Figure 26.

The results from the variographic characterisation showed that RC sampling exhibit less sampling variability than manual BH sampling, which in turn showed lower variability then the segment sampling method, Figure 26. The nugget-to-sill ratio was also lower for RC sampling, indicating that this method is superior to the BH sampling methods in regard to sampling reproducibility.

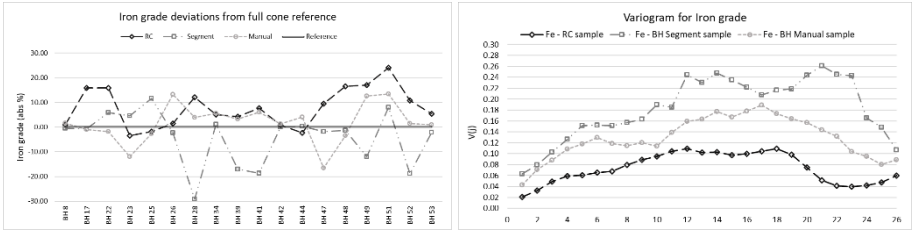

Figure 26. Left: comparison between the drill sampling methods and the full cone reference results, right: variogram for iron grade for the evaluated drill sampling methods (paper III).

This study concluded that the heterogeneity of the ore to be sampled is of critical importance to allow for proper discrimination between sampling methods. The results from the case studies showed that the large in-situ heterogeneity also affects the twin hole comparison, even though the hole distance was only 1.5m. In the specific mining conditions in Leveäniemi open pit mine, manual BH sampling was deemed fit-for-purpose for the blending of ore in to the primary crushers. Even if RC sampling was found to be more precise than the BH sampling, the increase in drilling cost and resources needed in the field and in-house laboratory could not be motivated based on the results of this study alone.

Summary of open pit mine sampling

The overall conclusion related to the open pit mine sampling studies, is that it is not possible to determine one universal best practice sampling approach for all open pit mine drill sampling situations. The wide variety of ore body characteristics, ore compositions and heterogeneities, local mining conditions and drill rigs around the world, means that the choice of drill sampling approach need to be determined in relation to local conditions.

There are advantages and disadvantages with both BH- and RC sampling. Both sampling methods are accompanied with various problems, but with correct identification and counteraction, they can often be reduced to reach fit-for-purpose representativity. The conclusion in this study (paper III) is that for the mining situation in Leveäniemi, RC sampling exhibit lower sampling variability than the two evaluated BH sampling methods, Figure 27. However, the RC sampling was not deemed sufficiently superior to justify the increased costs for drilling and sample handling.

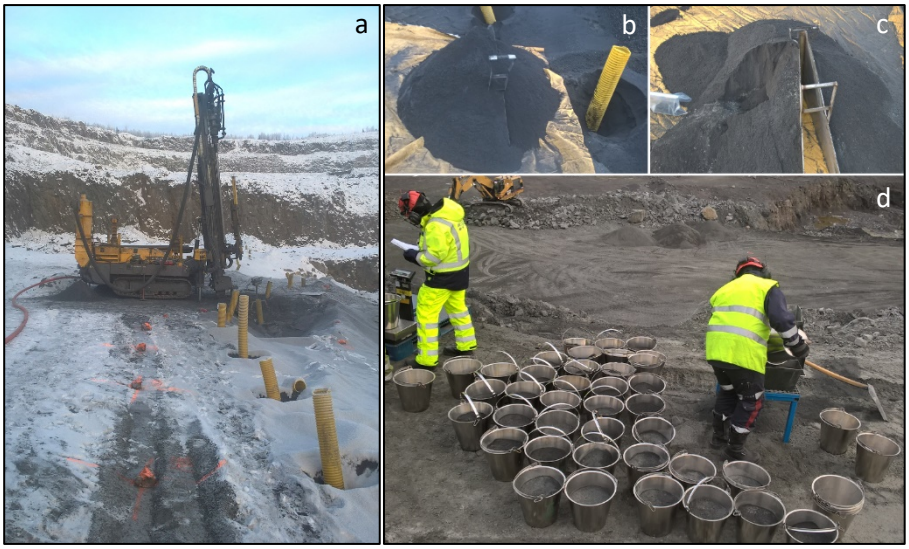

Figure 27. a) RC drilling during variographic experiment (paper III), b) sectorial sampling frame in BH cone (paper II), c) extracted manual BH sample and segment BH sample during variographic experiment (paper II), d) weighing and sample division during full cone BH reference sampling (paper II).

As no general best practice can be determined for open pit mine sampling, this PhD study concludes that the specific heterogeneity characteristics of the mine need to be understood to enable selection of a drill sampling method appropriate for the present ore body characteristics and mining conditions. A well planned variographic sampling experiment, along a profile of relevant ore heterogeneity, is one example of a powerful method for characterisation of both ore heterogeneity and sampling variability for the applied drill sampling procedure. This approach allows mining geologists, mine planners and process engineers to evaluate the specific measurement system performance in direct relation to the local mining conditions and ore characteristics.

3.2.2 Environmental recipient water sampling (paper IV)

The environmental recipient water sampling at LKAB is in many ways different from the other sampling processes studied within this PhD project. The recipient water sampling is performed outside of the actual mining and processing value chain and is not used for process or quality control. Nevertheless, high performance of these measurement systems is at least as important, as the recipient water analyses are critical for LKAB to assess and take responsibility for the environmental impact from the iron ore operations. Furthermore, they are also essential to retain the necessary environmental permits from the Swedish government. As the requirements for control of measurement uncertainty and total measurement system variability has increased, a comprehensive evaluation of the recipient water sampling processes was completed and is presented in paper IV of this thesis.

Paper IV: Experimental evaluation of surface water sampling variability for environmental monitoring in iron ore operations using concepts from the theory of sampling (TOS)

There has traditionally been a lack of focus on the sampling variability in environmental monitoring, while the requirements for certified and well documented analytical practices are strict. However, an increased scientific focus on sampling variability is an incentive for the industry to be proactive by including the evaluation of sampling processes in the mandatory self-monitoring programs related to environmental analysis. This study was performed as a pilot study to assess sampling variability at two important surface water sampling targets at LKAB, and to assess applicable methods for evaluation of measurement system performance. Duplicate- and replication experiments were performed at the two sampling targets KVA and SVA, affected by LKAB operations in Kiruna and Svappavaara respectively. Both the current and an alternative sample extraction method (using the Aloha sampler™) was evaluated through these experiments.

Duplicate experiments were performed at both sampling targets, taking the ambiguity in the two different sample protocols into account. The sampling protocol for KVA specifies a week for extraction of a sample once a month, while the SVA sampling target is sampled twice a week. The duplicates at KVA were randomly spaced between one and six days, while the SVA duplicates were collected in direct repetition during the same sampling round, to reflect the ambiguity described by the two sampling protocols. Duplicate analysis was applied for the primary samples extracted at KVA, to allow for estimation the analytical precision. The results showed that the KVA sampling variability was significantly higher than the SVA variability for all evaluated parameters. These results indicate that the temporal spacing between duplicates has a large impact on the estimated sampling variability. The relative measurement variability was below 20% for all analysed parameters at SVA, while the majority of parameters at KVA showed measurement variabilities between 30% and 80% (alkalinity, conductivity and phosphorus excluded). These results indicate that the measurement system variability is acceptable under repeatable conditions, or as described by a strictly defined sampling protocol. However, large temporal ambiguity in a sampling protocol may lead to excessive measurement variability due to the temporal heterogeneity of the stream water.

The replication experiments, Figure 28, were aimed at evaluating the current sampling procedure as well as the alternative sampling equipment named Aloha sampler™. The results showed a relative sampling variability of less than 20%, indicating acceptable sampling performance for both sampling methods for all analysed parameters. The Aloha Sampler™ showed slightly higher sampling variability than the current grab sampling method, for all parameters except alkalinity. This is likely due to that the Aloha Sampler™ covers a larger part of the cross-stream heterogeneity and collects increments during longer periods of time than the current method. The reason that this did not increase the measurement variability for alkalinity is probably a response to the lower overall variability for this parameter, compared to all other analytes.

Figure 28. Replication experiment of recipient water sampling results in a large number of samples for the environmental laboratory (paper IV).

An important conclusion in this study was that the ambiguity in the sampling protocol will have a large effect on the sampling variability and hence needs to be accounted for when designing experiments for evaluating measurement system performance. Duplicate and replication experiments, designed to fully cover the ambiguity in the sampling protocol, was found to be practical and rewarding methods for both initial and regular quality assessment of recipient water sampling. To allow for a complete evaluation of the sampling variability, representative for all seasonal variations in flow and analyte concentrations, the experiments should be applied continuously and regularly over the whole year.

The recipient water sampling procedures at LKAB were concluded to be acceptable under repeatability conditions for both the current sampling procedure and the Aloha Sampler™. However, the temporal ambiguity of routine samples at KVA leads to a measurement variability of above 20% for several parameters. A sampling solution, that can account for the large temporal heterogeneity of the stream water, is an automated sampler collecting time or volume proportional increments to be combined to composite samples for analysis.

3.2.3 Process sampling (papers V, VI and VII)

Paper V, VI and VII of this thesis concern evaluation and optimisation of various measurement systems in the mineral processing value chain at LKAB. These studies focus on two aspects; i) specific sampling systems where evaluation and improvements have been requested or problems have been identified, and ii) the general application of variographic characterisation to allow objective analysis and quality assessment of measurement system performance.

Paper V: Evaluation of sampling systems in iron ore concentrating and pelletizing processes – Quantification of Total Sampling Error (TSE) vs. process variation

This study focuses on applying variographic characterisation to a selection of measurement systems in the concentrating and pelletizing plants in LKAB iron ore operations in Kiruna. The aim was to evaluate the total measurement system variability to form a basis of suggestions of possible improvements. A general study objective was also to exemplify the application of variography in a mineral processing industry to illustrate the possibilities for improved quality control of both process and measurement system variabilities.

The results from the study showed that the measurement system variability for determination of iron grade after primary milling constitutes approximately 80% of the total variability for the manual grab sampling method applied. The variogram for iron grade of magnetite slurry showed acceptable low nugget-to-sill ratio of 3-5% for the primary shark-fin sampler and automated sample preparation and XRF analysis. This low sampling variability contradict general TOS understanding as the shark-fin sampler is accompanied with both IDE and IEE that normally introduce sampling bias that increase the measurement system variability. For some time periods, the measurement system for determination of iron grade of magnetite slurry indicated a periodicity of around 8-10h. The source of this specific cyclic behaviour could not be identified, but likely reasons are milling fluctuations, changes in crude ore, or flushing of the sampling system.

The manual grab sampling for moisture determination of filtered slurry followed the TOS anticipation of high sampling variability due to the significant ISE introduced by the incorrect sample extraction. The nugget-to-sill ratio of 60% indicated a non-acceptable precision leading to low possibilities for correct control of the filtering process. The linear cross stream sampler extracting iron ore pellets for automated size determination showed a nugget-to-sill ratio of 25-30%. This could be assessed as slightly high, but as the main reason for this is the low overall variability visible in the sill, the measurement system is deemed fit-for-purpose.

The conclusion from this study was that the large amount of process data readily available, enables the application of variographic characterisation at regular intervals in the LKAB mineral processing value chain. This allows for quantification of sampling and process variability as well as evaluation of measurement system performance. The application of empirical studies also proved its purpose as one presented example transgressed conventional expectations, in that a shark fin sampler was found to have acceptable nugget-to-sill ratio. This study supports previous suggestions that significant benefits can be obtained by applying variographic characterisation to continuous industrial processes.

Paper VI: Improvement of sampling for moisture content analysis in iron ore pellet feed – Variographic characterization and on-line IR-analysis

Paper V deemed the manual grab sampling (belt sampling) for moisture content determination of filtered slurry, i.e. iron ore pellet feed in plant 1, as non-representative. As a result of this conclusion, an in-depth study was initiated to evaluate alternative approaches to optimise the measurement system for moisture content determination. This study included further evaluation of the current belt sampling in plant 1, and evaluation of a composite spear sampling method applied in LKAB plant 2, an alternative composite filter sampling method, Figure 29, as well as on-line Infra-Red (IR) analysis.

Figure 29. Manual collection of a composite filter sample (paper VI).

Variographic characterisation was applied to long term process data for the current belt sampling method as well as detailed experimental data for both the belt sampling and filter sampling methods. The current belt sampling method exhibited a high nugget-to-sill ratio of 60% for the process data. The variographic experiment for the same sampling method showed a variogram which in practical interpretation was considered flat, i.e. the measurement system variability covers all process variability visible in the sill. The variogram for the experimental filter sampling data showed a nugget-to-sill ratio of 25%, i.e. indicating an acceptable relative measurement system variability. The filter sampling method collects three full-depth increments from three points of the filter discharge, but not a complete cross section of the material. However, the low nugget-to-sill ratio of the variogram indicate that the induced IDE and IEE are sufficiently reduced to achieve a fit-for-purpose measurement variability. The current spear sampling method and the IR analysis employed in plant 2 were also evaluated using variographic characterisation. The results showed that both methods exhibit similar nugget effects and sill levels as the filter sampling method tested in plant 1. A large advantage of the IR analysis, compared to all other methods, is the rapid analytical response. However, non-trivial calibration problems have been encountered, meaning that this method is not readily applicable for process control.

The conclusion of this study was that filter sampling is the preferred method for moisture determination as it leads to significantly lower sampling variability and simultaneously enable control of individual filters. This is particularly useful for correct adjustments of single filters, for example after filter cloth changes, as well as for detection of unexpected shifts or problems in individual filters. Recommended further studies include automation of the filter sampling method as well as evaluation of IR or other on-line analysis applied to each filter separately, to achieve real time analysis as well as process control of individual filters.

Paper VII: Total process and measurement system characterization using variography – covering the complete ore-to-shipping value chain at LKAB

Process and quality control in the LKAB mining and processing value chain is based on a variety of measurement systems. The objective of the study in paper VII was to evaluate all measurement systems throughout the LKAB processing value chain. The aim was to decouple measurement variability from true process variability to enable comparison between measurement systems and to focus attention where improvements are most needed to enable optimisation of measurement system performance at LKAB.

The overall results showed that the majority of the studied process measurement systems exhibit low total variability, indicating stable sampling processes. However, detailed inspection did reveal measurement systems in need of improvements. Three out of the 34 evaluated measurement systems stand out with variograms exhibiting significantly larger variabilities than the rest. Two of these three variograms represent sampling of final products in the pelletizing plant and at ship loading respectively, with subsequent on-line size determination. These two variograms are increasing, with a low nugget-to-sill ratio, indicating that the linear cross stream sample extractions and automated sieving analyses are fit-for-purpose for describing the large variabilities seen in the pellet size fractions being analysed. The third high-sill variogram represent sampling and iron grade analysis of tailings in the sorting plant. This variogram was close to flat, with a nugget-to-sill ratio of 70%. For more detailed evaluation of this measurement system, a variographic experiment was performed with a decreased time-spacing between analyses. The variogram for the experimental data showed a nugget-to-sill ratio of 35%, indicating that the ISE associated with the manual grab sampling of tailings cannot be adequately reduced with a smaller sampling interval. As the iron grade analysis of tailings is a critical measurement for assessing the performance of magnetic separation in the sorting plant, this measurement system is prioritised for improvements. A pilot study was therefore initiated to evaluate the possibility of using on-line PFTNA technique to increase the measurement system performance.

The study in paper VII also showed medium-sill variograms with significantly different appearances. For example, the 4h analysis of KH45 of green pellets showed a nearly flat variogram, the 24h silica grade analysis of iron ore pellets showed a continuously increasing variogram, Figure 30, and size determination of iron ore pellets showed an increasing variogram levelling off at the range of 12 hours for 5-9mm, while the variogram for 12.5-16mm is indicating a periodicity, Figure 30.

The results from this study clearly showed the strength of empirical evaluations. Some results supported general TOS knowledge, e.g. that cross belt hammer samplers and manual grab sampling introduce significant sampling errors that inflate the measurement variability. However, the study also identified both shark fin samplers and spear samplers (i.e. TOS-incorrect sample extraction methods) with sufficiently low nugget-to-sill ratios to be validated and deemed fit-for-purpose. These results indicate that the sampled material is

sufficiently uniform and that the ISE accompanying the shark fin and spear samplers were not excessively affecting the measurement variability.

A general conclusion presented by paper VII was that to enable continuous measurement system monitoring with the use of variographic characterisation, automated data collection and variogram generation is essential. Variographic characterisation was shown to reveal important information regarding the representativity of the employed measurement systems and the possibility for continuous quality control of these system is a desirable improvement compared to the current visual inspections. The calculation of variograms enable objective evaluations, allowing rational prioritisation of where measurement system improvements can have the largest cost-benefit effect.

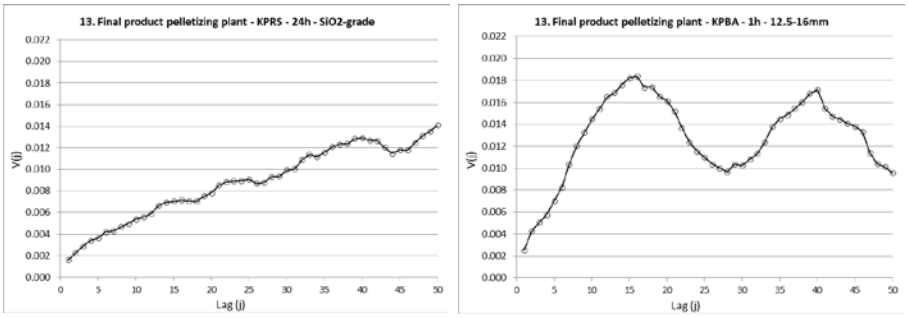

Figure 30. Two examples of the medium-sill variograms presented in paper VII.

Summary of process sampling

The application of variographic characterisation to available process data is a powerful tool for decomposition of variabilities stemming from the measurement system and the process respectively. This PhD study has shown that variography is able to characterise the performance of measurement systems in all parts of an iron ore processing value chain. It is readily applicable to the abundant existing process data and has the ability to reveal additional information if applied to specific high-resolution experimental data. However, to reach the full potential of variographic characterisation, automated data collection and variogram calculation is required. This would allow for continuous control of measurement systems, similar to process control charts, which are updated in real time.

One of the most valuable contributions of variography is the possibility for empirical evaluation and factual based assessment of measurement system. This is an important addition to the expertise of research and process engineers, to enable objective evaluations and conclusions regarding the performance of measurement systems. There is a need for numerical evidence to allow for correct prioritisation and decision making regarding measurement system improvements as well as quantification of the improved level of variability with an exchanged measurement system.

3.3 General conclusions and discussion

Representative sampling processes are essential for controlling iron ore mining and minerals processing operations efficiently. The representativity of samples and measurements is crucial for operators and engineers to be able to optimise process performance and to reach correct quality of final products. Reliable measurements are important for precise process control and to achieve both high productivity and low energy consumption. Furthermore, representative sampling processes are a critical requirement to ensure that results from pre-feasibility studies are valid and thereby lead to correct decisions.

This study concludes that there is no single general best practice for open pit mine drill sampling that can be consider superior in all applications. The results rather indicate that the representativity of a particular sampling approach is strongly related to local ore characteristics, heterogeneity, mineralogy and to the current mining conditions. This means that empirical pilot studies of the specific ore heterogeneity in the open pit to be sampled is essential to determine appropriate drill sampling methods and conditions in each case. The empirical results from the present studies indicate that manual BH sampling is locally fit-for-purpose for blending in to the primary crushers in the studied Leveäniemi open pit mine. RC sampling is able to lower the variability stemming from the sampling process, but the increased precision was not deemed to outweigh the considerable extra drilling costs and the additional resources needed for sample handling, preparation and analysis.

Variographic characterisation of measurement systems has been applied to several sampling targets in the LKAB mining and processing value chain. This study concludes that variograms provide valuable information about several different sampling situations, both in open pit mine drill sampling and in process sampling. For further, in depth evaluation, high resolution variographic experiments extracting samples with smaller spacing than normal procedures, can be applied advantageously to specific measurement systems. Replication and duplicate experiments have also been shown to render important information about measurement system performance. These methods are especially useful in situations where variography is not applicable due to for example restricted data availability. However, to allow correct evaluation and facilitate valid conclusions from sampling experiments, it is important to cover all temporal or special heterogeneity aspects in the process and sampling targets under evaluation. One of the most important benefits with all three experimental methods is the possibility for objective evaluation of measurement system performance. Traditionally, evaluation of sampling systems relies on visual inspection as well as engineering knowledge and assumptions, rather than empirical analysis. The possibility to use historical and on-line process data and/or specific experiments for variogram calculation, and hence discrimination of variability contributions, is valuable to allow for correct decision making based on numerical evidence. This is valued by process and research engineers as it allows for impartial evaluation of the performance of sampling systems, which is required for justifying investments related to improvement or replacement of, for example costly primary sample extraction methods and/or equipment.

The application of variographic characterisation has been shown to provide reliable, objective and transparent conclusions in the evaluation of measurement system performance in iron ore operations. However, manual variogram data collection and manual variogram calculation involves an extensive effort for e.g. data acquisition and handling of missing data. Therefore, this study concludes that to enable comprehensive and continuous measurement system evaluation through the entire process value chain, automated data collection together with automated variogram calculations would be required. This could for example be arranged so that variograms and variogram characteristics are updated regularly and presented in suitable control charts. By establishing a target level for the variogram characteristics, changes in process and/or measurement variabilities could be identified through the application of standard control limit approaches.

This PhD study has clearly shown that the application of empirical studies is rewarding in order to determine the performance of a given measurement system implementation. Conventional wisdom and general conclusions can only tell so much about which methods are appropriate or fit-for-purpose in specific, local contexts. Thus, the final verdict of how well a measurement system is able to perform can only be made after empirical evaluation of the system in its implemented situation and applied to the lot material that it is routinely used for. This study has several times shown results that contradict conventional TOS expectations. Both manual BH sampling and shark fin slurry sampling has for example been proven to have low nugget-to-sill ratios, i.e. low relative measurement system variability, leading to acceptable sample precision. Simultaneously, other empirical studies have supported general TOS conclusions, for example regarding cross belt (hammer) samplers and manual grab sampling as being non-representative. In summary, each measurement system and sampling approach need to be evaluated on its own merits, preferable with appropriate empirical methods as for example variographic characterisation, duplicate or replication experiments, to allow objective assessment of its performance.

3.4 Future work

This study has evaluated a large number of industrial measurement systems in LKAB iron ore operations in northern Sweden. The results regarding accuracy and precision have been varied and various conclusions, partly surprising and partly as-expected, have been reached. A series of recommendations have been presented in the appended papers and some pilot studies have already been initiated.

Examples of specific recommendations for future studies include:

i) Application of automated BH sampling equipment connected before the dust collector cyclone on the D65 drill rig used for BH drilling in LKAB open pit mines.
ii) Empirical heterogeneity characterisation of ore deposits to enable correct selection of appropriate drill sampling methods and parameters.
iii) Regular duplicate or replication experiments of recipient water sampling, taking the ambiguity in the sampling protocol into account.
iv) Implementation of recipient water sampling protocols and/or automated sampling that counteracts the temporal stream water heterogeneity.
v) Automation of filter sampling method for moisture content determination of iron ore pellet feed.
vi) Improvements of the IR-analysis for moisture content determination of iron ore pellet feed to reduce the calibration problems currently encountered.
vii) Use of a recirculating system for sampling of the fine fraction after primary milling in the concentration plant.
viii) Implementation of a continuous variographic characterisation facility to allow for comprehensive quality control regimes for all important measurement systems.

For some of the sampling systems evaluated in this study, further pilot studies have been initiated to implement new measurement systems that are able to improve accuracy and/or precision. One example is the iron grade of both incoming ore and tailings in the sorting plant, where the pilot study involves PFTNA technology that use neutron activation to excite the atoms of the material. The gamma rays from the excited atoms are detected and used to quantify the elemental content of the material. Furthermore, a study where the aim is to use machine learning to predict the moisture content of iron ore pellet feed, based on the abundant availability of various process data, has been initiated. Another possibility to improve this measurement system could be to apply image analysis of the filter cake to predict the moisture content of each separate filter. These are interesting potentials for new involvement of chemometrics and advanced IT approaches.

One of the most important recommendations for further research and development concerns an automated software/system for data acquisition and variogram calculation. To enable continuous quality control of the performance of measurement systems through the use of variographic characterisation, manual data logging or extraction for calculation of variograms is not possible in a large processing industry as LKAB. Automated generation of variograms, with features such as effective handling of large data sets and a rational

approach to define, detect and resolve issues regarding missing data and outliers, together with determined target values and tolerance limits for variogram characteristics, would make comprehensive and real-time assessment of measurement systems throughout the entire processing value chain possible. Furthermore, to allow for valid and rapid detection of deviations in process or measurement system variability, investigation and development of methods for statistical weighting of data in relation to age could be of significant interest.

As a complement to the variographic approach applied on traditional (sample and analysis) measurement systems, it would be favourable to also incorporate other process data, e.g. flows, temperatures, tank levels, pressure and power consumption into a measurement monitoring system. This would give more comprehensive data input and hence a possibility for faster detection of deviations in the process or measurement system variability, as each measured value can be compared to overall and historical process behaviours. By identifying and modelling patterns for expected measurement results under normal operating conditions, it should be possible to detect significant measurement and/or process deviations (true outliers) and enable identification and statistical estimation of the most probable root causes. In order to achieve a generic method and reduce manual calculations, modern machine learning methods could possibly be of interest.

The aim of the recommended further research is to optimise the quality of measurement data presented to mine geologists, mine planners, process engineers, plant operators and research engineers, for improved process monitoring and decision making. This is essential to allow for optimal control of all processes in the mining and processing value chain, as well as reaping the full potential and reach valid conclusions from feasibility studies and dedicated research projects. There is a significant carry-over potential from the present scope and results to several other process industry sectors. The present, as well as future results from the scope of investigations started with this thesis, will be presented at designated international conferences and published in appropriate scientific journals.

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PART 2

Appended papers

Paper I

A comprehensive literature review reflecting fifteen years of debate regarding the representativity of reverse circulation vs blast hole drill sampling

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TOS Forum (2017) 7:36-46
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A comprehensive literature review reflecting fifteen years of debate regarding the representativity of reverse circulation vs blast hole drill sampling

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Blast hole sampling is widely used for grade control by the mining industry all over the world, both in precious and base metal open pit mining. Blast hole (BH) samples are often regarded as inferior in comparison to "proper drill sampling" like reverse circulation (RC) and diamond (core) drilling (DD), and are accused of lacking representativity by the sampling community. The present paper aims at collecting all peer reviewed publications from 2000 onwards that concern open pit mine sampling performance of BH, RC and/or DD drill sampling. This will form a comprehensive literature review reflecting on the debate between the representativity of the different sampling methods. The literature review collected a total of 31 publications (two were more or less duplicates and one consisted of an abstract only). The main source for publications on RC and BH drill sampling were dedicated sampling conferences, other mining conferences and some publications were found in peer-reviewed journals. From the gathered publications, it is not possible to draw a general overall conclusion as to the superiority of one drill sampling method over another. Both RC and BH have advantages and disadvantages and the choice of system needs to be related to the ore type and to the mining conditions. The overall conclusion is that it is always necessary to evaluate the specific sampling system to be used in light of the Theory of Sampling (TOS) (and with respect to the characteristics of the ore to be mined). It is always necessary to ascertain that the specific drilling sampling system contemplated does not lead to hidden losses that could have been avoided or missed profits that could be gained with a more relevant and representative sampling system. It would appear that the mining industry is doomed to continue to follow local, often economy-driven objectives and sampling solutions even if these can be documented as inferior when seen in the light of the representativity imperative. A call is made for universal adherence to the principles laid down by TOS for representativity in the primary sampling stage, before economic, logistical or other (local) factors are allowed to intervene. What is the objective to analyse and to make decisions in the mining industry, based on samples that can be documented not to be representative?

Introduction

In the mining industry, misclassifications of ore types due to poor sampling practices can easily generate large value losses and contribute to economic inefficiency in the crushing stages, as has been vividly demonstrated by Carrasco *et al.*[1] Internal calculations at LKAB indicate that misclassification of ore can lead to unnecessary costs of up to US$200,000 if one blast of waste is classified as ore, or loss in revenue of up to US$700,000 if one blast of ore is classified as waste. These estimates only represent pure costs or losses, and do not include losses due to decreased quality of final products, loss of customer trust, increased product handling or increased strain on waste dumps and dams. These examples clearly show the need for correct and representative sampling methods in open pit mining, for high quality and cost effective mining operations.

Blast hole (BH) sampling is widely used for grade control by the mining industry all over the world, both in precious and base metal open pit mining. BH samples are often regarded as inferior in comparison to "proper drill sampling" like reverse circulation (RC) and diamond (core) drilling (DD) and are accused of lacking representativity by the sampling community.[2,3] Figure 1 presents some of the well-known BH sampling problems and issues. Nevertheless, many mining operations continue to rely on manual BH sampling methods which are claimed to lead to "good results". However, Abzalov *et al.*[4] concluded in a study of (mainly) existing BH and RC samples in

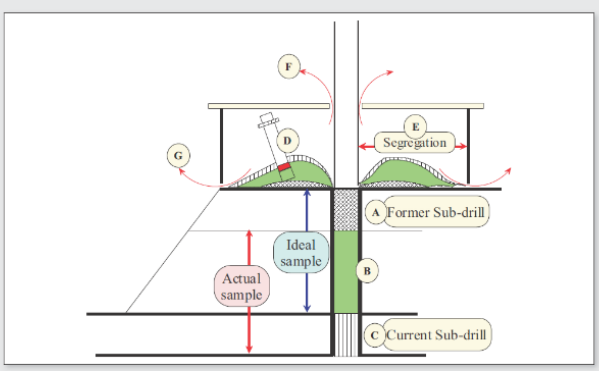

Figure 1. Summary of blast hole sampling problems and errors (from Reference 2 with permission).

Figure 2. Manual BH sampling a. a LKAB open pit mine. a. Cutting a sectorial part of the BH cone b. Collecting a vertical slice of uniform thickness from the bottom to the top in the centre of the blast hole cone.

an iron ore deposit, that both methods can be equally biased compared to full cone BH sampling. See Figure 2 for a contemporary example of manual sampling in iron ore open pit mining.

The present paper aims at collecting all peer reviewed publications from 2000 onwards that concern open pit mine sampling performance of BH, RC and/or DD drill sampling. This will form a comprehensive literature review reflecting on the debate between the representativity of the different sampling methods. With a summary of published conclusions the authors will attempt to see if it is possible to find an overall consensus regarding the superiority of any of the sampling methods.

Method

This literature review is conducted with both a quantitative and qualitative focus. First, all identified papers covering the topic of open pit mine drill sampling performance were compiled in a complete reference list. The search for publications was done through the web-based databases SCOPUS and ScienceDirect with keywords: "blast hole (BH) sampling", "open pit mine sampling", "drill sampling" and "reverse circulation (RC) sampling". The search also covered review of proceedings from the specific sampling conferences Sampling and World Conference in Sampling and Blending, as well as proceedings from various mining conferences and congresses. Last, all references in the hitherto gathered publications were reviewed for any further publications on the topic. Apart from internet based searches, some physical digging was also conducted, Figure 3.

The abstracts of all primary identified publications were reviewed to collect articles that specifically discuss the representativity or performance of at least one of the three drill sampling methods. Publications that concern a drill sampling method, but do not further discuss its representativity or precision of collected samples were excluded from the literature review during review of abstracts. The focus of the literature review is to assess performance, i.e. representativity and/or precision of open mine drill sampling methods; all publications that discussed this issue were included in the review. As the performance of actual drilling, *in situ* or bulk sampling or assay methods is not the main focus, publications that only discuss these matters were excluded from the review. Publications regarding underground drill sampling have also been excluded from the review as the present focus is on open pit mine sampling.

The ambition from the authors was to identify all publications from 2000 and onwards addressing the representativity of drill sampling methods. There might, however, still be some publications that could not be identified with the search methods used here. Any additional literature items that may surface in this context will be included in an updated survey which will be the base for discussions in the PhD thesis which includes the present feature. Defence is planned for 2019.

Results

The main sources for peer reviewed publications in the subject of open pit mine sampling and its representativity were specific sampling conferences, i.e. *Sampling* in Australia and the international *World Conference in Sampling and Blending* biannual series. A second source is other mining conferences and a few publications could be found in peer-reviewed journals. See Table 1 for the sources of all publications in the literature review. Comprehensive references for all publications can be found in the list of references. A brief summary of the most important conclusions from all collected publications can be found in Table 2.

The collected publications include seven theoretical discussions based on the Theory of Sampling (TOS) as well as previous publications and personal experience. In 22 of the publications, one or more case studies were conducted to evaluate the performance of one or both drill sampling methods. See Table 2 for a summary of all publications. Two publications were more or less identical with the same case study, results and conclusion; consequently, only one has been added to this summary.

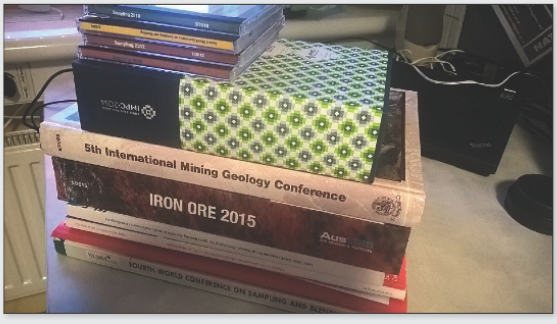

Figure 3. A selection of some physical sources collected for the literature review.

Table 1. Sources for the collected publications.

Publication	Sampling specific conference	Other mining conference	Peer reviewed journal
Total: 31 publications	16	10	5
Abzalov *et al.*[4]		✓	
Abzalov *et al.*[5]			✓
Alfaro[6]	✓		
Caccioppoli *et al.*[7]		✓	
Carrasco *et al.*[1]			✓
Chieregati *et al.*[8]	✓		
Chieregati *et al.*[9]	✓		
Chieregati *et al.*[10]	✓		
Crawford *et al.*[11]		✓	
El Hajj *et al.*[12]	✓		
François-Bongarçon[13]	✓		
Goers *et al.*[14]	✓		
Gomes *et al.*[15]	✓		
Hapugoda *et al.*[16]	✓		
Hapugoda *et al.*[17]		✓	
Holmes[18]			✓
Holmes[19]			✓
Hoogvliet[20]		✓	
Kirk *et al.*[21]		✓	
Magri *et al.*[3]	✓		
Magri *et al.*[22]		✓	
McArthur[23]	✓		
Minkkinen *et al.*[24]	✓		
Niemeläinen *et al.*[25]	✓		
Ortiz *et al.*[26]		✓	
Pitard[27]		✓	
Pitard[2]	✓		
Séguret[28]	✓		
Spangenberg *et al.*[29]			✓
Young[30]		✓	
Ziegelaar *et al.*[31]	✓		

Last, one publication consisted only of an abstract, the study was presented orally at a conference in full but no article was prepared for the proceedings. In 12 of the case studies, existing grade control data was used while in 16 publications experiments were performed to generate new data, Table 3.

Table 4 shows a summary of the drill methods evaluated, the reference method and the most important conclusion of each publication. Table 4 also show which ore type is mined in the case studies presented. About half of the publications evaluate BH sampling and the other half compare BH to RC sampling. Four of the publications only evaluate RC sampling. The most common reference method used is DD sampling (nine publications), while full cone BH sampling, RC sampling and plant feed or reconciliation are used as reference in three to six publications. In as many as seven publications, RC drill sampling is assumed to be representative by the author(s), either from previous publications or "by experience". At the same time five (other) publications conclude that RC sampling can be non-representative as evaluations show several sampling problems and biases. Eleven publications conclude that RC sampling is more representative then BH sampling, while thirteen publications indicate that BH sampling can be representative or fit-for-purpose.

In summary, the results and conclusions show a very diverse picture of the debate between RC and BH sampling. One weak indication *could be* that base metal mining (iron ore) might show a slight tendency to accept BH sampling as representative. In contrast, the literature review shows that for sampling in gold mining, RC is generally concluded to be more representative than BH sampling. One exception is Chieregati[8,9] which both conclude that BH sampling can be fit for purpose if using correct equipment and sampling procedures, Figure 4.

Discussion

The different aspects of RC vs BH sampling are complex and in all cases clearly relate to the specific ore type and the prevailing mining conditions. The wide range of conclusions from all publications show that there is no universal answer to one sampling method always being superior. BH sampling is indeed accompanied by many problems like loss of fines, upward/downward contamination, influx of sub-drill material, pile segregation, pile shape irregularities, operator-dependent sampling, too small sample size, frozen BH cones and non-equiprobabilistic sampling equipment, see Figures 5 and 6 and References 2 and 6 among others.

Solutions do exist that handle some of the problems related to manual BH sampling and are able to reach a representative or fit-for-purpose status, however. Examples that counteract the most glaring sampling bias problems are channel sampling and sectorial sampling, Figures 4 and 7. Another solution that has proved to provide representative BH samples (in two publications) is automated BH sampling systems, Figure 8. Even though these can produce good quality samples, they have not made a breakthrough on the market for BH sampling, mainly due to the increase in drilling time when applying the automated sampling approach.

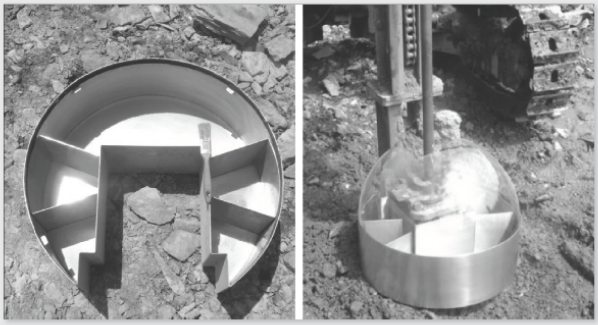

Figure 4. New machine sectoria sampler fitted to the AWH drill (right) and detail of the buckets/frame (left) (reproduced from Reference 9 with permission).

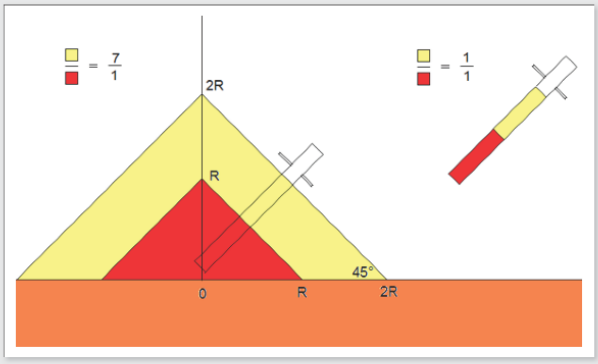

Figure 6. Non equiprobabilistic sampling tube (reproduced from Reference 8 with permission).

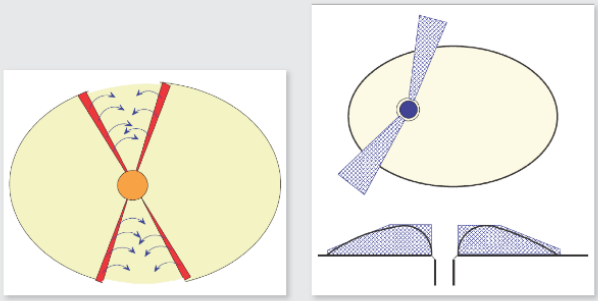

Figure 7. Left: digging two radial channels, from which to extricate four thin, radial increments to make a composite sample. Right: correct design and positioning of radial bucket/sectorial sampler. Reproduced from Reference 2 with permission.

Figure 5. Uneven fill piles are a big problem in some open pit mines, from Reference 6.

The scale of resolution of sampling grids is in many publications concluded to be more important than sampling performance. Typical RC sampling grids are *ca* 25×25 m compared to BH sampling grids that are in most cases around 5×5 m. This large difference in grid size often leads to a larger increase in the number of misclassified mining blocks than BH sampling imperfections. If great care is taken when developing sampling methods, adapted to the drill rig at hand and accommodating the need of each mining situation, BH sampling can come satisfactorily close to being fit-for-purpose in some mining situations. In other case studies, RC sampling is proved to be more representative and is proved

Figure 8. Top: Drillsampler™ from Harrison Cooper for automatic blast hole sampling installed underneath the drill deck (reproduced from Reference 2 with permission). Bottom: the Finnish "Autosampler" system with Soffoore™ sample socks attached (reproduced from Reference 24 with permission).

Table 2. Main conclusion of collected publications.

Publication	Main conclusion from publication
Abzalov *et al.*[4]	Manual BH sampling with shovel from BH cone on 6×6m grid, compared to RC drill sampling on 25×25m grid. BH and RC sampling proved equally biased in comparison to full BH cone assays. BH and RC or DD results are consistent at distances of 1m, but the variations in grade between twin holes increase when the distance increase and holes 10m apart show excessively poor repeatability. Indicating that a sampling grid of 25×25m will be sub-optimal at this mine. The quality of grade control procedures depends on both quality and quantity of grade control samples. In this case study, the amount of misclassified selective mining units would increase from approximately 5.8% to 12.3% when increasing sampling grid from 5×5m to 25×25m.
Abzalov *et al.*[5]	Comparison between BH and RC sampling in iron ore open pit mine. The study incorporates both sampling error and sampling grid to optimise sampling procedures. Currently, BH sampling is used for grade control, but RC sampling was considered as an alternative approach for grade control. Sample duplicates and twin holes with RC and BH sampling revealed that RC sampling does not guarantee improved sample quality. RC and BH exhibit similar precision errors and RC were biased, underestimating Al_2O_3 and SiO_2 grades, and overestimating Fe grades. Simulation showed that change to RC grade control with 25×25m grid would not reduce grade control errors, but rather increase the number of misclassified ore and waste blocks.
Alfaro[6]	A case study of the Rio Blanco ore deposit is porphyry copper, located in the central zone of Chile. Many problems with manual BH sampling in winter due to moist and frozen material. Comparisons between BH and DD assay results is done by identifying holes with maximum inter distance of 5m. The comparison indicates problems with the BH samples, especially for As and Mo.
Caccioppoli *et al.*[7]	Comparison between RC and manual BH sampling is done for flitch mining. The authors are using RC drilling as reference for the manual BH samples that are taken separately for top and bottom flitch. The result show differences between full cone BH and RC assay results in 20% of the blast holes. Improvement of the material recovery in the remaining blast holes could improve the accuracy of the BH assays.
Carrasco *et al.*[1]	A case study of BH sampling in a porphyry copper operation shows a nugget effect of 70% of the total variability. The sampling did not take the equiprobable rules into account and collected 250g of material from a 2ton lot with 2cm top size. The variability was much greater than for diamond drilling even though DD had a much smaller support. By the use of statistical and geostatistical calculations, the authors calculate losses due to poor BH sampling to approximately 22 MUSD.
Chieregati *et al.*[8]	Summary of several aspects that make sampling gold challenging based, on two case studies. Even though RC drill sampling is regarded as a more appropriate sample method, a significant (up to 20%) loos of fines can occur through overflow of the cyclone. BH sampling also have problems with loss of fines due to wind, and manual sampling with a shovel is common and does not conform to TOS equiprobabilistic principles. The authors suggest that the use of a correctly designed sampler could eliminate problems with delimitation, extraction and weighting errors in BH sampling. RC sampling can also be improved, for example by adding a secondary cyclone to collect the fines.
Chieregati *et al.*[9]	Validation of a newly designed cupola stationary sectorial sampler for BH sampling. The sectorial sampler has a significantly higher recovery of fine material which minimised sampling error due to loss of fines. The sampler did not lower production compared to previous manual BH sampling. The two opposite sectorial sample collectors were unbiased to each other. The sampler did not show any bias to the reference used, which in this case was TOS correct sampling of the plant feed. The authors note that double-discharge drills are a completely different scenario and cannot be directly compared to this case study with single-discharge, narrow diameter drill.
Chieregati *et al.*[10]	Case study of RC drill sampling compared with manual BH sampling. Complete BH cone as well as complete RC material was used as reference. Result show that the BH drilling loses coarse material in the hole and the manual BH method oversamples the coarse particles. The RC sampling system is unbiased compared to the complete RC material. The new RC rig shows both representative sampling results as well as increased reconciliation reliability.
Crawford *et al.*[11]	Investigation of manual BH sampling proved it to inaccurate and have poor repeatability. Trials showed that RC sampling was able to produce better sample representativity and depth flexibility, but the cost of RC was too high for the operation. The solution for improved BH sampling was to implement an automatic sampling system for the BH rig. This sampler could collect four samples over the 8m drill hole depth. Even though some loss of ultrafines resulted in a sampling bias, the flexibility and improvement of sampling representativity compared to manual sampling outweighed the concerns about ultrafines.

Publication	Main conclusion from publication
El Hajj *et al.*[12]	Manual sampling from a BH drill rig and RC sampling was compared to each other as well as to a DD reference. Both BH and RC sampling overestimate Au and Cu grades, but the bias for BH is about double to RC. The manual BH sampling is in turn underestimating the Au and Cu grade leading to satisfactory, but illusory, reconciliation results. Conclusion show that the manual BH sampling method was not suitable for reconciliation. There was also concluded that the estimate errors from the sampling method was not as significant as the errors from the type of drill rig used. Recommendations are to work with automated RC drill sampling in spite of extra cost and more traffic in the mine.
François-Bongarçon[13]	A comprehensive discussion of advantages and disadvantages of both BH and RC drill sampling without prerequisite assumption on one or the other's superiority. Conclusion states that the advantages and disadvantages are not clear-cut between the two methods, and certainly not as much as previously presented. Resolution is concluded to be a more critical factor than the performance of each sampling method as long as greatest of care is given to obtaining unbiased samples.
Goers *et al.*[14]	Three different RC drill sampling systems on two different drill rigs have been tested. The systems tested were: conventional cyclone and three tiered splitter sampling system, the Rotaport cone splitting system and the Prograd ex PGX1350R sampling system. Field duplicates and fines samples were collected to assess the sampling performance during the testing. Results show that the fines have a different grade then the rest of the material, meaning it is essential for the sampling system to sample the fines as well, which the PGX1350R managed. Field duplicates cannot alone be used to assess sample quality as loss of material from the drill hole or sampling systems is not detected. "With sample analysis costs of US$25–30 per sample and annual total drilling assay costs of US$1–1.5 M confidence that sample quality is high is critical. The efforts and costs to produce these high quality samples are justified with the knowledge that the downstream effects of poor samples and the decisions made from them can result in the loss of profits and increase in production costs."
Gomes *et al.*[15]	Case study of the mine to mill reconciliation including analysis of possible BH sampling biases. Comparison was made between manual BH sampling using a canvas and using a drum fitted to the drill, with small opening for the drill rod. Results show that the normal method resulted in a loss of fines as the mass of the drum sample was 8.7% greater and the relative mass of the two finest fractions were much larger than for the canvas method. The study led to development of a BH sampler with cupola that further improved sample representativity.
Hapugoda *et al.*[16]	Comparison of DD, RC and RAB drill sampling methods. Conclusion is that DD is able to produce the best samples but is expensive and slow. RC has a better sample recovery and provide reasonably uncontaminated samples compared to the RAB sampling. Some identified problems with RC include damaged pipes, excessive dust generation. Advantages of RAB drilling include lowest cost, greater speed and large sample volume.
Hapugoda *et al.*[17]	More or less the same article as above, published in a different forum. Identical evaluation, results and conclusion.
Holmes[18]	Theoretical discussion about problems and solution with lack of representativity for BH sampling. Recommendation include taking sectorial or radial cuts from the BH cone, either using some sort of sectorial cutters placed prior to drilling, or using a shovel after the drilling is finalised. Using an automatic sample divider on a cyclone that collects the drill cuttings is also recommended but has many problems like loss of material around the blast hole as well as loss of fines in the dust filter. RC sampling is mentioned as a recent advance for drill sampling but not evaluated for representativity.
Holmes[19]	Theoretical discussion about problems with BH sampling similar to above publication. RC drill sampling is presented as being considered best solution for open pit mine sampling even due to the much higher cost. Presented solutions for accepted BH sampling is extracting radial sectors, vertical slices or channel cuts from the BH cone. Another suggestion is automated collection of drill cutting using compressed air and a cyclone. Best approaches for BH are, however, considered to be channel sampling or sectorial cutters.
Hoogvliet[20]	A case study of a gold and silver mine in Borneo where the grade control system was changed from BH sampling to RC sampling. The original sampling method was to collect samples over 2.5 m using a wedged pie sampler at the collar of the blast hole, any existing sub-drill is not sampled. After viability studies showing improved profits, the grade control was changed to RC drilling. Reconciliation studies show that the annual profit increased by approximately US$2.87 M after implementation of RC. Even after deducting the extra cost for drilling, over US$2 M remained. The authors conclude that desktop studies comparing different drill sampling methods are not sufficient and often overestimate possible profits. The best method to evaluate a new method is by reconciliation and actual produced ounces, i.e. profitability. Another conclusion is that even if RC in some cases has a large impact on profitability, the benefits over BH sampling may be minimal in some other situations.

Publication	Main conclusion from publication
Kirk *et al.*[21]	Evaluation of BH sampling in regards to RC sampling and evaluation of implementation of RC drill sampling systems as a substitute for the BH sampling. BH samples were collected every 2.5m intervals using a wedge-shaped sampling tray placed radial to the drill string. Two previous studies had indicated that the BH sampling performed reasonably well for high-grade or and waste samples. For low- to medium-grade samples the BH sample was biased as the low grade mineralisation occurs predominantly in the fines that was lost in the BH drilling process and therefore not sampled. Some other BH sampling problems were high top size compared to sample size, as well as frequent collar collapses contaminating the samples. The main result of the biased BH sampling was that approximately 30% of low grade ore was misclassified as waste due to the loss of fines. The BH sampling was also showed to be less accurate than RC and DD sampling.
Magri *et al.*[3]	Theoretical simulation of the economic losses due to poor BH sampling as well as using kriging or polygonal estimation as estimation method. 10%, 20% and 30% fundamental sampling error for BH sampling was used in the simulation to approximate the losses. These numbers are derived from sample systems commonly used in the mining industry (not explained how). The study shows that both estimation methodology and sampling errors lead to losses of millions of dollars per annum.
Magri *et al.*[22]	Case study with comparison between BH and RC drill sampling as well as collection of complete BH cones. Duplicate samples from the BH manual sampling was also collected for analysis of precision. Results show that radial bucket BH sampling is biased compared to both complete BH cone and RC samples for $CaCO_3$. The study also compared previous results from DD, RC and BH which showed good correspondence between all methods and biases between BH and DD were lower then between BH and RC. Variograms were used to estimate nugget effect and these were very low for both RC and DD, but BH nugget effect was considerably larger in spite the larger support for BH. Conclusion is that "Higher quality samples and better short term planning could be achieved by replacing BH sampling with RC sampling, if an economic analysis which includes the hidden costs of misclassified blasted material supports the change."
McArthur[23]	Case study of manual BH sampling in flitch mining. Experiments were carried out to evaluate if the manual method to divide the BH cone in upper and lower flitch and sub-drill is representative. Result show large variability in the ratio between flitches and sub-drill causing problems when sampling. The sub-drill is over represented by an average of 10%. The conclusion is that despite the misallocation of some material between flitches and sub-drill, comparative assay result show that the manual sampling method produce and overall unbiased result.
Minkkinen *et al.*[24]	Case study of a new automatic sampler for BH drill rig. A sampling belt collects a sectorial sample from the drill cutting ejection and transfer the material to a rotating cone splitter. Full BH cone samples were used as reference and in general the new sampling method showed good agreement with this reference.
Niemeläinen *et al.*[25]	Test of an on-line XRF analyser for percussion surface drill rig. The conclusions are that the system is equally representative as DD and RC drill sampling, but much faster. Some deviations between results could be seen but is expected to come from calibration problems. The on line analyser does not collect the dust (similar to RC) as this is deviated by the dust collector.
Ortiz *et al.*[26]	The authors conclude in the introduction that BH samples have poor quality due to time and space constraints, that most BH sampling methods suffer from delimitation, extraction and segregation-related errors. The authors use a simulation methodology applied to three case studies to evaluate the performance of different sampling methods on different drilling grids. The relative error for BH sampling is evaluated by duplicate sampling to a range between 14% and 20% while the error for RC sampling is set from zero to 8%. The conclusion is that moving from BH to RC sampling provides significant economic benefits reaching millions of dollars per annum. "The case studies show that when operating conditions allow for a dedicated drilling rig, it is worth considering investing in a sophisticated sampling system mounted on an RC drilling rig to operate well in advance, thus providing timely data for building short-term models that can include several additional relevant variables."
Pitard[27]	Theoretical discussion regarding sampling, including RC and BH sampling methods. Problems with RC sampling is said to be down the hole contamination, preparation error, selective separation of coarse and fine particles and poor or excessive recoveries leading to extraction biases. BH sampling is presented as a monumental problem for the mining industry due to delimitation, extraction and preparation biases. The author also discusses three new automated BH sampling methods that are stated to be able to produce correct and representative samples. As the systems are not yet in production it is not clear which will be most reliable, but they do represent a major breakthrough in ore grade control for the mining industry according to the author.

Publication	Main conclusion from publication
Pitard[2]	Theoretical discussions about problems associated with BH sampling and advantages with RC drilling. Examples of BH sampling problems presenter are: upward/downward contamination, upward material losses, refluxing, sub-drill material, pile segregation, pile shape irregularities, loss of fines, operator dependent sampling, sampling interfering with mining productivity, too small sample size, vertical drill holes and so on. A few presented advantages with BH sampling are: the same drilling technique for blasting and grade control, small visible cost, good lateral interpolation, less traffic in the pit. Presented advantages with RC sampling are: absence of sub-drill, possibility to drill several benches at once and to drill at an appropriate angle, limited contamination and losses, no interference with productivity, can drill months ahead of mining, possibility to drill less but better holes, smaller sample mass, information from lower benches, better vertical definition of ore and waste, automation is easy, and so on. The disadvantages with RC presented are: additional visible cost, increase in traffic in the pit. The conclusions are that BH sampling cannot provide representative samples and that RC sampling provide many advantages that may far outweigh the additional cost.
Séguret[28]	Case study of a copper mine in Chile. Comparison between BH sampler and DD using 3000 DD samples and 13,000 BH samples for the study. The authors use vertical and horizontal variograms, migration and cross variograms to evaluate the sampling methods. Conclusion show that DD sampling has errors and that both DD and BH variograms show approximately 50% nugget effect. Analysis of the BH error leads to conclusion that it is not the primary sampling step that generates the error, but it can rather be found later in the process. The authors suggest that DD and BH are used together for short term mine planning and that linear systems can be used to remove nugget effect from the data.
Spangenberg *et al.*[29]	The authors state RC drilling as preferred open pit mine sampling with no discussion regarding BH sampling. The authors discuss a few aspects of RC splitters that are biased and should be avoided. A specific sample mass reduction solution is mentioned as being representative and therefore correct.
Young[30]	Case study of a Zn/Pb/Ag mine where traditional BH sampling was replaced by RC sampling. BH sampling was conducted by using a PVC pipe, collecting eight increments from the BH cone. Problems with this method include: cone destruction by rigs, hole vs sample number mismatch, incorrect sampling technique, vertical drill holes in 75° ore body and time constraints. The BH sampling is, however, stated to have been relatively reliable when blasting benches of consistent height. Implementing RC sampling instead of BH did not increase cost for samples handling as the drill grid increased but the samples per hole increased. However, the cost of drilling increased due to the dedicated sampling drill holes that are not drilled when sampling blast holes. Comparison between the sampling methods (using RC as reference) show that BH sampling misclassified 18% of waste as ore and 13% of ore as waste. The cost of processing this waste without cost of lost opportunity (ore going to waste) more than covers the cost of RC drilling.
Ziegelaar *et al.*[31]	This publication only has an abstract and no prepared article for the conference presentation. The study is a comparison of different drilling techniques with DD used as reference. No conclusions are given by the abstract.

Figure 9. Left: drilling operations using a conventional cyclone and three-tiered splitter system. Right: drilling operations using the PGX1350R sampling system. Reproduced from Reference 14 with permission.

Table 3. Context and data collection methods in publications.

Publication	Theoretical discussion	Case study	Use of existing grade control data	Experiment generating new data
Total: 31 publications	10	22	12	16
Abzalov *et al.*[4]		✓	✓	✓
Abzalov *et al.*[5]		✓		✓
Alfaro[6]		✓	✓	
Caccioppoli *et al.*[7]		✓		✓
Carrasco *et al.*[1]	✓	✓	✓	
Chieregati *et al.*[8]	✓	✓	✓	
Chieregati *et al.*[9]		✓		✓
Chieregati *et al.*[10]		✓		✓
Crawford *et al.*[11]		✓		✓
El Hajj *et al.*[12]		✓	✓	✓
François-Bongarçon[13]	✓			
Goers *et al.*[14]		✓	✓	✓
Gomes *et al.*[15]		✓		✓
Hapugoda *et al.*[16]		✓		✓
Hapugoda *et al.*[17]	More or less the same article as above, published in a different forum.			
Holmes[18]	✓			
Holmes[19]	✓			
Hoogvliet[20]		✓	✓	
Kirk *et al.*[21]		✓	✓	✓
Magri *et al.*[3]	✓		✓	
Magri *et al.*[22]		✓	✓	✓
McArthur[23]		✓		✓
Minkkinen *et al.*[24]		✓		✓
Niemeläinen *et al.*[25]		✓		✓
Ortiz *et al.*[26]	✓	✓	✓	
Pitard[27]	✓			
Pitard[2]	✓			
Séguret[28]		✓	✓	
Spangenberg *et al.*[29]	✓			
Young[30]		✓		✓
Ziegelaar *et al.*[31]	Abstract only, no paper was prepared for this presentation			

to generate a large increase in profit when substituting BH sampling.

One major concern that is widely addressed is that the cost of RC drill sampling is "too high". Even when improved sampling performance can be proved, the increased cost for RC sampling is typically not accepted by the mining operation. This is most often due to the fact that it is more or less impossible to exactly quantify the possible value gain or economic losses due to inaccurate BH sampling.

There are several mining operations using RC drill sampling for short-term grade control despite the higher costs. Especially in precious metals mining, the improvements with RC drill sampling have proven to result in larger profit increase than the cost of drilling.[20]

However, the conclusion regarding representativity of RC drilling is not uniform in all publications. Some publications state as a prerequisite that RC is representative, while others conclude that RC, just as BH sampling, can be proven to be biased. Goers[14] evaluates different RC drill sampling systems and concludes that the choice of sampling system for the RC rig as well as the complete system for RC sampling and handling determines if sampling can be representative. Loss of fines, leading to sample bias, is for example a major problem with some RC sampling systems, see Figure 9.

Conclusions

The literature review collected a total of 31 publications (two were more or less duplicates and one consisted of an abstract only). The main source for publications on RC and BH drill sampling were dedicated sampling conferences, other mining conferences and some publications were found in peer-reviewed journals.

From the gathered publications, it is not possible to draw a general overall conclusion as to the superiority of one drill sampling method over another. Both RC and BH have advantages and disadvantages, and the choice of system needs to be related to the ore type and to the mining conditions. The overall conclusion is that it is always necessary to evaluate the specific sampling system to be used in the light of TOS (and with respect to the characteristics of the ore to be mined). It is always necessary to ascertain that the specific drilling sampling system contemplated does not lead to hidden losses that could have been avoided, or missed profits that could be gained with a more relevant and representative sampling system.

It would appear that the mining industry is doomed to continue to follow local, often economy-driven objectives and sampling solutions even if these can be documented as inferior when seen in the light of the representativity imperative. A call is made for universal adherence to the principles laid down by TOS for representativity in the

Table 4. Scope and reference methods used in publications.

Publication	Evaluation of BH sampling	Evaluation of RC sampling	Comparison between RC and BH sampling	Full BH cone used as reference	RC drilling used as reference	DD used as reference	Plant feed or reconciliation used as reference	Prerequisite assumption of RC as being representative	Conclusion show RC to be more representative than BH drill sampling	Conclusion show that RC drill sampling methods can be non-representative	Conclusion show RC and BH drill sampling to be equally (non) representative	Conclusion show that BH sampling can be representative / fit-for-purpose	Conclusion show that BH sampling is not representative	Sampled ore used to draw conclusion
Total: 31 publications	12	4	15	6	5	9	3	7	11	5	5	13	6	
Abzalov *et al.*[4]			✓	✓		✓				✓	✓	✓		Fe
Abzalov *et al.*[5]			✓							✓	✓			Fe
Alfaro[6]	✓					✓							✓	Cu, Mo
Caccioppoli *et al.*[7]			✓		✓			✓	✓					Fe
Carrasco *et al.*[1]	✓					✓							✓	Cu
Chieregati *et al.*[8]	✓	✓									✓	✓		Au
Chieregati *et al.*[9]	✓						✓					✓		Au
Chieregati *et al.*[10]			✓	✓	✓			✓						Au, Cu
Crawford *et al.*[11]			✓	✓					✓			✓		Fe
El Hajj *et al.*[12]			✓	✓		✓		✓						Au, Cu
François-Bongarçon[13]			✓							✓	✓	✓		—
Goers *et al.*[14]		✓			✓					✓				Au
Gomes *et al.*[15]	✓						✓					✓		Au
Hapugoda *et al.*[16]			✓			✓			✓					Au, Cu
Hapugoda *et al.*[17]	More or less the same article as above, published in a different forum.													
Holmes[18]	✓											✓		—
Holmes[19]	✓								✓			✓		—
Hoogvliet[20]			✓				✓		✓					Au, Ag
Kirk *et al.*[21]			✓			✓			✓					Pt
Magri *et al.*[3]	✓						✓						✓	Au, Cu
Magri *et al.*[22]			✓	✓		✓			✓					Cu
McArthur[23]	✓											✓		Fe
Minkkinen *et al.*[24]	✓			✓								✓		Fe
Niemeläinen *et al.*[25]			✓		✓	✓					✓			Cu, Ni
Ortiz *et al.*[26]			✓					✓	✓				✓	Au, Cu
Pitard[27]	✓	✓								✓		✓	✓	—
Pitard[2]			✓					✓	✓				✓	—
Séguret[28]	✓					✓						✓		Cu
Spangenberg *et al.*[29]		✓						✓	✓					Au
Young[30]			✓		✓			✓	✓			✓		Zn, Pb, Ag
Ziegelaar *et al.*[31]	Abstract only, no paper was prepared for this presentation													

primary sampling stage, before economic, logistical or other (local) factors are allowed to intervene. What is the objective to analyse and to make decisions in the mining industry, based on samples that can be documented not to be representative?

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Paper II

Blasthole sampling (replicate and variographic experiments) in LKAB open pit iron ore mines: fit-for-purpose representativity?

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Blasthole sampling (replicate and variographic experiments) in LKAB open pit iron ore mines – fit-for-purpose representativity?

K Engström[1,2] and K H Esbensen[3,4,5,6]

ABSTRACT

The conclusion from the debate over the last decade in the mining sector is that, for grade control, reverse circulation (RC) drill sampling gives more reliable results due to less sampling problems than blasthole samples. However, by conducting the latter with well-controlled procedures there still exist fair possibilities to counteract the major blasthole sampling problems and achieve fit-for-purpose representativeness, especially if based on minimal loss of fines. In the present PhD study, extensive replicate experiments and variographic characterisations are conducted to evaluate the performance of manual blasthole (BH) sampling and sectorial segment sampling. The experiments were performed in LKAB's Leveäniemi open pit mine in the north of Sweden. The experimental area is an apatite-magnetite iron ore deposit of the Kiruna type, with the demand to exhibit a maximum of local heterogeneity to allow the experiment the largest possible conclusion powers. Gathering and splitting the complete BH drill cutting cones (fines cone, coarse cone) served as an authoritative reference on which to assess the representativity of the two BH sampling methods.

Results show that manual BH drill sampling is able to produce samples with an acceptable accuracy and precision if performed properly and with insight. Based on variographic comparison, segment sampling shows similar precision for chemical grades as the manual sampling, while the particle size distributions were more deviating for segment sampling than for manual sampling at the high and low size ranges. Possible reasons for these partly surprising results are discussed.

For further investigation of the performance of BH versus RC drill sampling, a second experiment has been planned on a parallel profile. This will result in an unprecedented opportunity for a full variographic comparison between RC and BH sampling, aimed at designing specific, fit-for-purpose sampling procedures as a function of ore quality, cut-off grades and important contaminant levels.

INTRODUCTION

The conclusion from the debate over the last decade in the mining sector is that, for grade control, reverse circulation drill sampling gives more reliable results and less sampling problems than blasthole samples (BH). It is claimed that this should be a motivation for accepting the associated higher drilling costs (Pitard, 2008; Magri, Magri and Neira, 2011; Chieregati *et al*, 2015). However, by conducting blasthole sampling with well-controlled correct sampling procedures in accordance to Theory of Sampling (TOS), there still exist fair possibilities to counteract the major BH sampling problems, actually being able to achieve fit-for-purpose representativeness with minimal loss of fines (François-Bongarçon, 2010; Chieregati *et al*, 2011, 2015). There only exist a few studies and theoretical research papers that have discussed the full scope of advantages versus disadvantages of RC versus BH sampling, with the notable publication of Pitard (2008, 2009). Focused experiments with the purpose of evaluating and comparing these alternative methods in base metal mining are difficult to find in publications. Two recent attempts are Minkkinen *et al* (2015) and Chieregati *et al* (2015), but each study only deals with one approach and does not compare the two.

With the current (2015) low metal prices impacting negatively on the mining industry, many companies struggle

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to lower production costs while at the same time increase productivity. This situation introduces a fundamental desire to optimise production as far as possible in every stage from mine to product. One important parameter in this quest is only to process ore with correct grades and never load waste rock into the primary crushers. The economic situation also adds focus on the need for precise quality control already in the open pit. The only way to make correct classifications of ore versus waste in the pit is by representative sampling procedures. Misclassifications of ore types, due to poor sampling practices, can easily generate large value losses and economical inefficiency in the crushers as has been vividly demonstrated by Carrasco, Carrasco and Jara (2004). Internal calculations at LKAB indicate that misclassification of ore can lead to futile extra costs of up to US$200 000 if one blast of waste is classified as ore, or loss in revenue of up to US$700 000 if one blast of ore is classified as waste. These estimates only represent pure costs or losses, but do not include losses due to decreased quality of final products, loss of customer trust, increased product handling or increased strain on waste dumps and dams. The need for more efficient and economic mining and refining processes increases the need for reliable pit sampling for short-term process planning. This has generated a strong focus on production drill sampling and its performance in mining operations around the world.

Blasthole (BH) sampling is widely used for grade control and short-term planning in both base metal and precious metals open pit mining. BH samples are often regarded as inferior with comparison to 'proper drill sampling' like reverse circulation (RC) and diamond (core) drilling (DD) and is accused of lacking representativity by the sampling community (eg Pitard, 2008; Magri, Magri and Neira, 2011). Nevertheless, many mining operations are still using manual BH sampling methods with claimed good results. Evaluations of the performance of individual BH sampling methods are rarely published and dedicated experiments with the main purpose of evaluating sampling performance are close to non-existent. Abzalov *et al* (2007) concluded in their study of (mainly) existing BH and RC samples in an iron ore deposit, that both methods can be equally biased compared to full cone BH sampling. According to simulations, a suggested increase in sampling grid with RC would result in an increased number of misclassified block values (Abzalov *et al*, 2007).

The present study evaluates current manual BH sampling methods as well as the possible sectorial segment BH sampling alternative (Chieregati *et al*, 2011) at LKAB. Extensive variographic and replicate experiments (RE) DS 3077 (2013) are used to enable a complete and extensive evaluation of both sampling methods. The experiments were constructed to give maximum conclusion power with a view to quantify the sampling precision of both current and alternative BH sampling methods in the Leveäniemi open pit mine. For further investigation of the performance of BH versus RC drill sampling, a second variographic experiment has been planned on a parallel profile. The PhD study will evaluate this complement of RC drill holes, positioned as close to the BH holes as physically possible (spaced on average 1.5 m between the two profiles). RC drill results will be published in a later publication due to the production timetable for these proceedings.

Experimental study objectives

The study objective is to evaluate the existing manual sampling method, as well as the possible segment sampling alternative at LKAB's open pit mines. The evaluation focuses on relative sampling variability (RSV, see DS 3077, 2013) and variographic comparison with due consideration to the possibilities for bias elimination in practical routine sampling. The experiments include the complementary RC drill results currently being processed.

LKAB'S LEVEÄNIEMI OPEN PIT MINE

The Leveäniemi open pit mine is located in Svappavaara, northern Sweden. The bedrock at Leveäniemi consists of metamorphosed rocks of volcanic and sedimentary origin, which locally are intruded by granite, pegmatite and diabase (Bergman, Kubler and Martinsson, 2001). These metavolcanic rocks, mainly of trachyandesitic in composition, are similar in age (1.88–1.91 Ga) and composition to the Porphyry and Porphyrite groups in the Kiruna district some 40 km to the north-west (Eilu, 2012).

While earlier regarded to be of sedimentary origin, the current understanding sees the Leveäniemi deposit as being of magmatic origin, supported by the detailed structural relationships outlined below (breccia versus intense veining). This re-interpretation sees the veining as a late-stage aspect of the magmatic ore genesis, which in itself is inferred to be a late differentiation feature of the andesitic volcanism, followed by later stage metamorphism and alterations as well as local structural faulting affecting the open pit geology to some degree.

The deposit is dominated by massive magnetite ore but in the central part of the deposit, magnetite is largely altered to hematite and martite. Large volumes of 'ore breccia' with mostly distinct contacts to the massive ore occur in an up to 100 m wide peripheral zone in the surrounding biotite schist. Apatite, calcite and amphibole are the main gangue minerals in the deposit while titanite, diopside, biotite and chlorite are less important (Bergman, Kubler and Martinsson, 2001). The massive magnetite as well as magnetite veins/breccias in the trachyandesites and biotite schist is the main ore type mined in Leveäniemi. In the pit it is observed how the deposit is bounded by faults, locally overprinted by shear zones.

The geological 3D model and resource block model in Leveäniemi are based on exploration diamond drilling carried out by Geological Survey of Sweden in 1957–1962 (Bergman, Kubler and Martinsson, 2001) and by LKAB in the 2000s. The experimental drill profile investigated in this study is likely to encounter a diverse set of complex primary lithology with different degrees of secondary alterations and may occasionally cut representatives of the subvertical granite/pegmatite/diabase veins. As it turned out, the sampling experiment certainly met the aim of a profile of significantly varying ore grades. It can be expected that the 55 BH and RC drill holes of 20 m depth will allow a significant test of the validity of the alternative sampling methods to be compared (Wild, 2015).

Blasthole sampling at LKAB

For short-term grade control and mine planning manual sampling from BH drill cones is currently carried out. Manual samples are extracted with a shovel from every second blasthole cone, covering the total vertical face of the axial centre of the cone (see Figure 1). The chemical assay results from the BH samples are incorporated in the resource block model to complete the sample grid for short-term production planning. It is observed that the ore in Leveäniemi is relatively easily fractured during BH drilling. The brecciated, intensely veined ore and the grading biotite schists are notably friable, which makes for a relatively fine-grained BH material that is easy to sample.

The blastholes are drilled with an Atlas Copco D65 BH rig which is equipped with a dust collector (for occupational

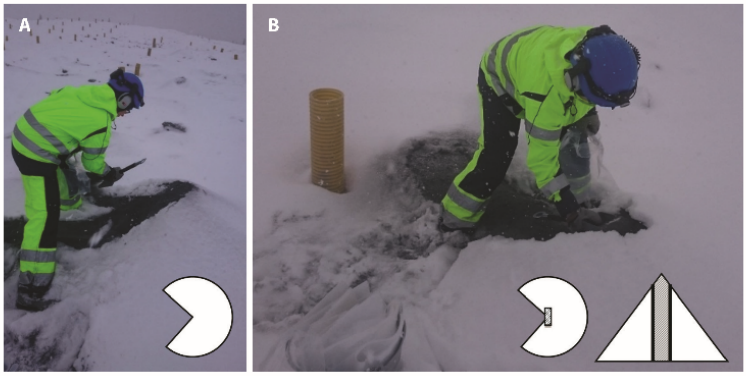

FIG 1 – Manual blasthole sampling at the Leveäniemi mine. (A) Cutting a sectorial part of the blasthole cone; (B) collecting a vertical slice of uniform thickness from the bottom to the top in the centre of the blasthole cone.

health and environmental reasons). The dust collector cyclone ejects the dominating part of the drill cuttings in the front of the drill rig while a smaller amount of finer material is ejected under the back of the drill rig. After drilling is completed, currently one sample from every second blasthole is extracted and sent to the laboratory for chemical analysis to enable ore classification for production planning. During the current manual sampling protocol, these field samples are only collected from the coarse front BH cone. ie the fine fraction is deliberately excluded from sampling – a clear violation of the Fundamental Sampling Principle (FSP), DS 3077 (2013).

During the manual sampling, a sectorial part of the BH cone is cut away using the shovel as illustrated in Figure 1a. When the axial centre of the cone is completely exposed and freely accessible, the field sample is collected as a vertical slice of uniform thickness taking equal amounts of material from each height of the cone (see Figure 1b). This sampling is always done from the bottom to the top of the cone. The representativity of the current manual BH sampling method is questioned by the mine geologists, which has initiated this study. From the perspective of TOS it would appear that this sampling scheme is not necessarily representative due to likely incorrect delimitation error (IDE) and possible incorrect extraction errors (IEE) (Gy, 1979; Pitard, 2004a, 2009). This is especially so relative to the sectorial BH cone sampling approach employed in many mining operations (Chieregati *et al*, 2011). However in the present study, the manual sampling approach used in Leveäniemi will be evaluated empirically together with its sectorial alternative.

For grade control in the Leveäniemi open pit, iron and vanadium as chemical parameters are used for short-term mine planning. For the present experiment however we also focus on silica as this is an important parameter in the following refining and pelletising process monitoring and control. Lastly the complete particle size distribution will be evaluated in order to optimise the discrimination power of the comparisons between the sampling methods.

REPLICATION EXPERIMENT

A replication experiment (RE) of current manual BH sampling was performed and evaluated according to Esbensen and Wagner (2016), DS 3077 (2013). The samples were collected from eight BH cones in three of LKAB's open pit mines resulting in a total of 24 BH cones. From each cone, six to eight replicate manual samples were collected, each as a full slice from the centre of the cone as shown in Figure 1b. It was not always possible to collect all eight planned samples from the more unstable cones which would have made the cones collapse precluding true replicate sampling. The minimum number of replicated primary samples was six, with the majority reaching the planned eight. All samples where dried and split into one subsample for size analysis, and a parallel for chemical analysis. Based on the number of realised replicate samples/analytical results, the relative coefficient of variance (CV_{rel}) was calculated for both chemical assays and particle size analysis according to Equation 1. As mentioned DS 3077 (2013) if the sampling procedure is not correct, ie sampling bias exist, the RE quantification of CV_{rel} will include effects arising from the variable sampling bias if not completely eliminated.

$$CV_{rel} = 100\left(\frac{\sigma}{\bar{X}}\right) = RSV \quad (1)$$

CV_{rel} relative coefficient of variance
σ standard deviation of the replicated analysis results
$\bar{X}$ average of the replicated analysis results
RSV relative sampling variability (relative sampling and analysis standard deviation)

The RE performed on the primary manual sampling will result in a RSV including all activities from primary sampling to analysis of the analytical aliquot. To be able to separate the variability originating from sampling, sample preparation and measurement, the RE has been repeated after primary sampling and after the sampling preparation procedures (hierarchical replication experiment). This will result in one CV_{rel} for the complete sampling system: RSV, one CV_{rel} for the sample preparation and analysis system: RPV (relative preparation and analysis variability) and one CV_{rel} for the analysis: TAE (total analytical error) allowing assessment of where the majority of the empirical sampling variability originates.

Replication experiment – results

The results from the RE were evaluated in regards to iron, vanadium and silica grade as well as for accumulated size fractions <4.0 mm, <1 mm and <63 µm. The estimated RSV

ranges from 0.38 per cent to 6.41 per cent for iron grade, from 0.58 per cent to 7.37 per cent for vanadium grade and from 0.36 per cent to 38.6 per cent for silica grade (see Figure 2), which displays the empirical RSV levels as a function of BH material grade, ie the average of the replicated analyses in each individual RE.

The RSV results show a clear trend for chemical analysis in that samples with low analytical grade have a higher RSV (entirely as expected). Areas in the open pit with higher iron grade (massive magnetite ore) are in general less heterogeneous which will result in a sampling situation generating less variability, ie a smaller RSV. In contrast, the ore with lower iron grade is more brecciated and therefore there is a greater challenge to collect representative samples for these ores. There are a few exceptions from this general trend, a result from samples collected from drill holes with large variations in iron grade in different parts of the drill hole, for example as shown in Figure 3.

The RSV for the key variables iron and vanadium is universally below eight per cent, with an average around four per cent. This is a surprisingly good reproducibility for the manual BH cone sampling approach (DS 3077, 2013). Up to eight replicated primary manual BH samples differ only between themselves to a degree ~5 per cent (relative). For silica, a few results show an RSV of up to 25 per cent and 38 per cent, but the majority of the calculated RSV's are less than ten per cent. This is consistent with the general geological understanding of the complex lithology encountered along the experimental profile as described above.

For the rich ores in Leveäniemi (iron grades of 50–70 per cent Fe) the particle size assays show a trend with lower RSV for ore with higher iron grade. The size analysis was only performed for Leveäniemi samples and therefore only eight REs were evaluated. All replicates indicate and RSV under eight per cent which is a strong statement that the reproducibility of the manual sampling method is quite acceptable in practice (Esbensen and Wagner, 2016). TOS puts great emphasis on particle size analysis results when evaluating the degree of accuracy and reproducibility.

The RPV was calculated to an average of 1.0 per cent for iron, 4.3 per cent for silica, and 1.1 per cent for vanadium grade. The trend for RPV was similar to the one shown by RSV, indicating that higher iron grade resulted in lower RPV and vice versa. The TAE has also been evaluated at the chemical and physical laboratory and is likewise presented in Table 1.

Even though a few isolated samples showed large RSV for silica, the overall results from the RE, for the key parameters iron, vanadium and grain size distribution appear to indicate that the manual BH sampling method is performing well in regards to sampling reproducibility. This finding is notable when it is factored in that all residual sampling bias effects are included (Gy, 1998; Pitard, 2004a; Esbensen and Wagner, 2016).

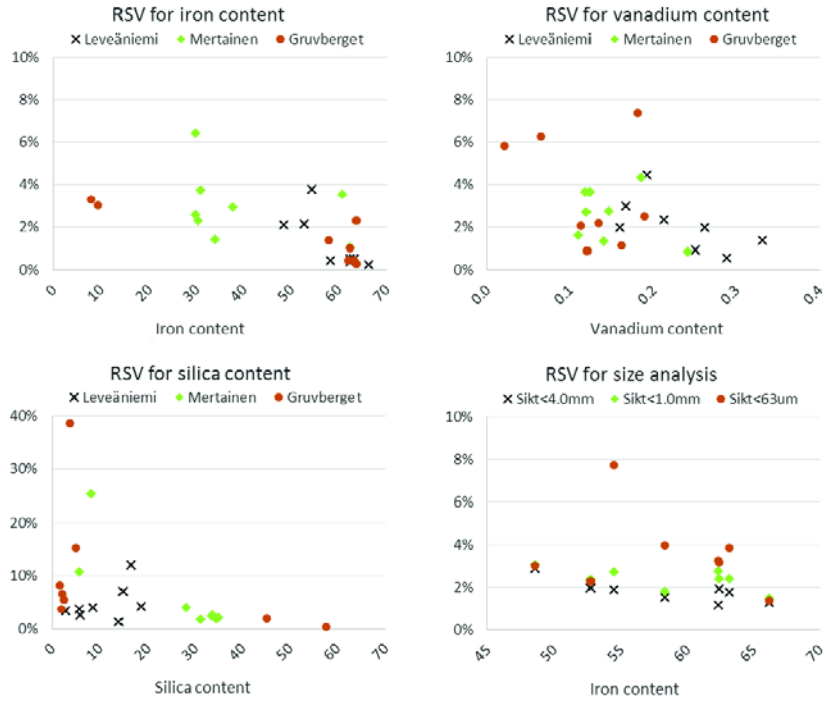

FIG 2 – Relative sampling variability (RSV) results from primary sampling replication experiments for iron, vanadium and silica grade from LKAB's three open pit mines, as well as RSV for size analysis (only from Leveäniemi open pit mine).

FIG 3 – Blasthole cone originating from drill hole with large variations in iron grade at different depths of the drill hole. The white material is rich in silica, while the black material is rich in iron. See text for a detailed geology description of the Leveäniemi ore and gangue.

TABLE 1

Average relative sampling variability (RSV), relative preparation and analysis variability (RPV) and total analytical error (TAE) from the manual sampling replication experiments performed in LKAB open pit mines. RSV includes all variable sampling bias effects.

(Relative standard deviation)	RSV	RPV	TAE (Fe > 50%)	TAE (Fe < 50%)
Iron grade	2.0%	1.0%	0.1%	0.5%
Silica grade	5.0%	4.3%	1.7%	0.8%
Vanadium grade	2.9%	1.1%	0.8%	1.0%
Size fraction <4.0 mm	2.5%	-	2.9%	
Size fraction <1.0 mm	4.4%	-	2.9%	
Size fraction <63 µm	8.0%	-	3.6%	

VARIOGRAPHIC EXPERIMENT

The variographic experiment transect is located in the north-west part of LKAB's Leveäniemi open pit mine. The transect was selected so that the sampled ore would exhibit potentially large variations in both chemical composition and mineralogy. This to enable highest possible conclusion power to the experiment as sampling variable ore is always more complicated and often generates larger sampling variability. The experimental transect is presented in Figure 4. The BH drill plan included 55 × 20 m deep holes, drilled 2.5 m apart along on a straight path in the open pit mine floor (140 m in length). This allows

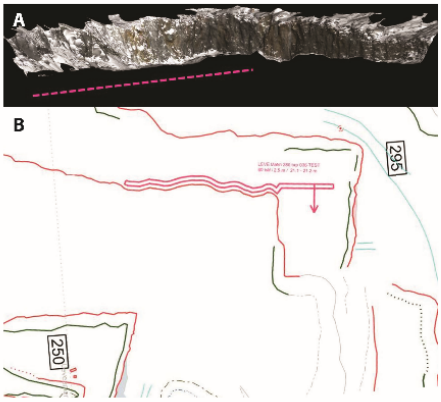

FIG 4 – (A) Mine block wall (220 m long) in Leveäniemi pit, located immediately west of the experimental transect that is shown as a dashed line. Note the conspicuous occurrences of fault lines and shear zones (see geological description). White areas are snow and ice; photograph November 2016, courtesy of mine geologist Celine Debras. (B) Location of experimental transect (140 m long) on level 280 in north-eastern part of Leveäniemi open pit mine. Arrow is indicating blast direction of experimental transect towards the middle/lower part of the pit.

for proper variographic characterisation of high-quality assay quantifications under controlled conditions.

The parallel B11 versus RC[7] variographic sampling experiments were conducted simultaneously as drilling progressed along the transect. Current BH sampling procedures are in contrast conducted after drilling is finalised. Ground canvases were placed under both drill rig material discharge outlet chutes, one canvas under the coarse discharge in the front and one under the fines discharge (see Figure 5). Under the coarse discharge, a sectorial cutter (see Figure 5) was placed on top of the canvas before drilling commenced. This sectorial cutter collected what is termed segment sample in this experiment, as illustrated in Figure 6.

FIG 5 – Experimental drilling with canvas placed under the back discharge and canvas plus sectorial cutter placed under front discharge of the Atlas Copco D65 drill rig.

7. RC drill results are to be published elsewhere due to the production timetable of these proceedings.

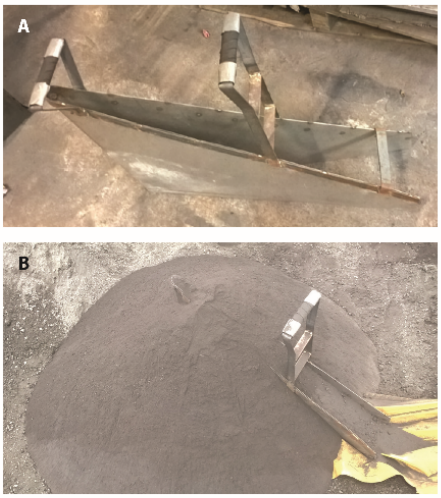

FIG 6 – (A) Sectorial sample cutter for delimitation and extraction of 'segment samples'. (B) Sectorial segment cutter at work in a typical blasthole coarse particles cone. A canvas is placed under the cutter to separate the blasthole material from the pre-existing ground material.

After drilling was completed, the manual sample (see Figure 1) was collected from the coarse BH cone, ie according to normal sampling procedures. Simultaneously, the segment sample was collected from the sectorial cutter (see Figures 6–7). Subsequently, all the remaining material from the coarse BH cone was collected, weighed and split into a primary coarse cone BH sample (see Figure 8). All the material from the fines cone was also collected, weighed and similarly split into a complementary primary fines cone BH sample. This procedure thus resulted in a total of four samples from each blasthole: the conventional manual sample, the alternative segment sample and the pair of complete coarse cone and fines cone samples. The analytical result for the complete BH material is calculated as the weighted averages of the results from all four samples. In the present experiment these 'total BH' analytical results serve as the authoritative reference to which the manual and the segments samples are compared. The first author was supervising the entire experimental drilling from A–Z. Great care was taken during drilling operations to avoid subdrilling effects.

FIG 7 – Manual and segment sample collected from a coarse blasthole cone.

FIG 8 – Complete coarse cone and fines cone were collected in buckets, weighed and split in the field.

Variographic experiment – results

The variographic experiment resulted in a total of 55 drilled blastholes, 2.5 m apart along the profile from which manual and segment samples were collected, accidental loss of material reduced the total number of evaluated drill holes to 53. (The vacuum collector for drill cuttings failed during drilling of hole number 54, resulting in a loss of all BH material so that no samples could be collected. As a consequence of this sample loss, BH number 55 was excluded from the analysis as the distance between holes 53 and 55 was twice that for all other holes.) A synoptic overview of the chemical characteristics of the manual samples from the 53 experimental blastholes is presented in Figure 9. Spikes with high silica in the chemical characterisation are due to granite veins common in the deposit. The large ore variations along the experimental transect furthers a strong conclusion power to the experiment as the comparisons made will cover practically all ore grade variations in the mine.

From 18 out of these 55 holes, randomly distributed along the transect, all BH material was collected in addition to the manual and segment samples (see Table 2). The complete coarse cone and complete fines cone were collected, weighed and split to produce two separate primary samples. The analysis results from all four collected samples were used to create a weighted average as a reference 'total' for each of these 18 holes.

Comparisons of the manual and segment sample to the authoritative reference are presented in Figure 10. The results indicate that for the most important elements; iron, vanadium and silica, the manual samples show least deviations from the total weighted average. The chemical grade of the fines cone is strongly deviating from the coarse cone, especially for hole numbers 47–53. Even though the mass of the fines cone is smaller (on average 25 per cent out of the total mass) than the coarse cone (on average 75 per cent of the total mass) the large difference in analytical results does affect the total weighted average significantly.

Even though the manual sample has the least deviations from the total result, the difference is significant for some of the sampled drill holes reflecting the ever present heterogeneity issues. Blastholes numbers 49–53 show a larger variation between the various sampling methods then all other holes. The most distinct difference at this end of the experimental transect is that the fines cone shows a lower iron grade then the coarse cone. This indicates that the mineralogy in this area strongly affects the breakage of the drilled material so that

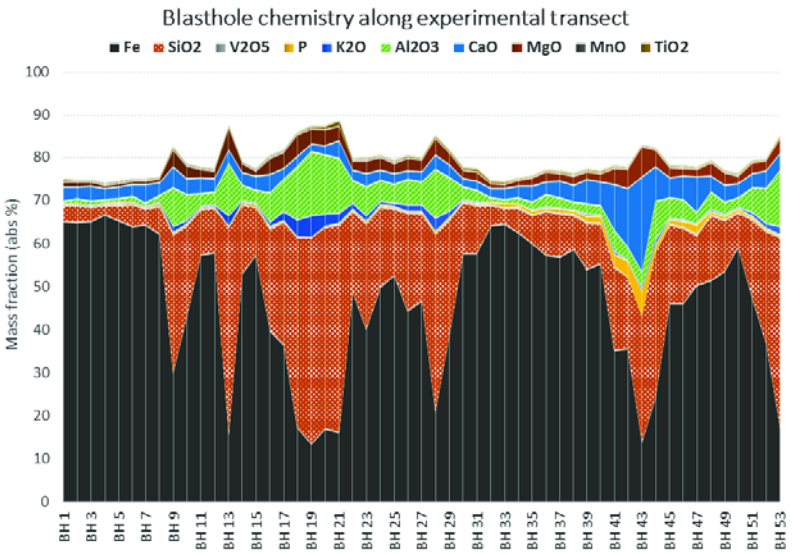

FIG 9 – Chemical composition of the manual blasthole samples along the experimental transect, documenting a highly variable ore composition. (The remaining percentages to reach 100 per cent are the oxides bound to the iron. The X-ray fluorescence analysis does not calculate the iron oxide grade but rather the free iron grade.)

TABLE 2

Identification of sample types collected from each blasthole along the experimental transect: 1 – manual sample, 2 – segment sample, 3 – coarse cone, 4 – fines cone, 5 – replicate experiment of coarse cone.

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53
1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
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iron-rich minerals primarily reside in the coarser material and vice versa for silica-rich minerals.

The essential evidence shown in Figure 10 is that the manual samples are in fact the closets proxies to the reference samples. Notable exceptions from this observation are the blastholes number 47 and 51, which does not support for the generality of the findings. There is however a higher number of 'irregular' deviations for the segment samples than for the manual counterparts. The main trend among all holes presented in Figure 10 is that holes with low heterogeneity, ie where the difference in grade between coarse and fines cone is small, the manual sample method is fit-for-purpose representative in comparison to the authoritative reference. In areas where the heterogeneity is larger and the grade differs between the fine and coarse fraction, the manual sampling exhibits larger deviation from both reference as well as the coarse cone from where it is extracted. This is not surprising as the mass of the manual sample is so small in comparison to the total mass, therefore the large heterogeneity in the material will increase the manual samples bias and precision. The key issue in this study will still be the variographic comparison between the manual and the segment samples which is presented in Figure 11.

For the two key variables iron and vanadium, the variographic characterisation shows that the along-profile variability for manual sampling is very similar to that for segment sampling (see Figure 11). If anything the segment samples show a slightly larger variability for higher lags. Both parameters delineate a range of ~5 lags (corresponding to 12–13 m) and exhibit largely similar sill levels (both variograms show a slight rise at larger lags, which is not considered significant compared to the surprising similarities). It is well known from the mine geology that iron and vanadium are strongly correlated in the Leveäniemi ore, as both reside in magnetite. For both these elements, the nugget effect (also called minimum possible error (MPE), eg Gy, 1998; Pitard, 2004a) is roughly corresponding to ~20 per cent of the sill level indicated at the range (see Table 3). This is interpreted as reflecting an acceptable total sampling error (TSE) for both field sampling methods (DS 3077, 2013). By contrast, the variogram for silica is very different showing a significantly higher nugget effect and sill, consistent with its independent geological and geochemical origin. There is no need for a specific interpretation of the silica variogram for this reason.

The main result of the variographic comparison is – rather against conventional expectations – that the supposedly inferior manual sampling appears to be at least equally

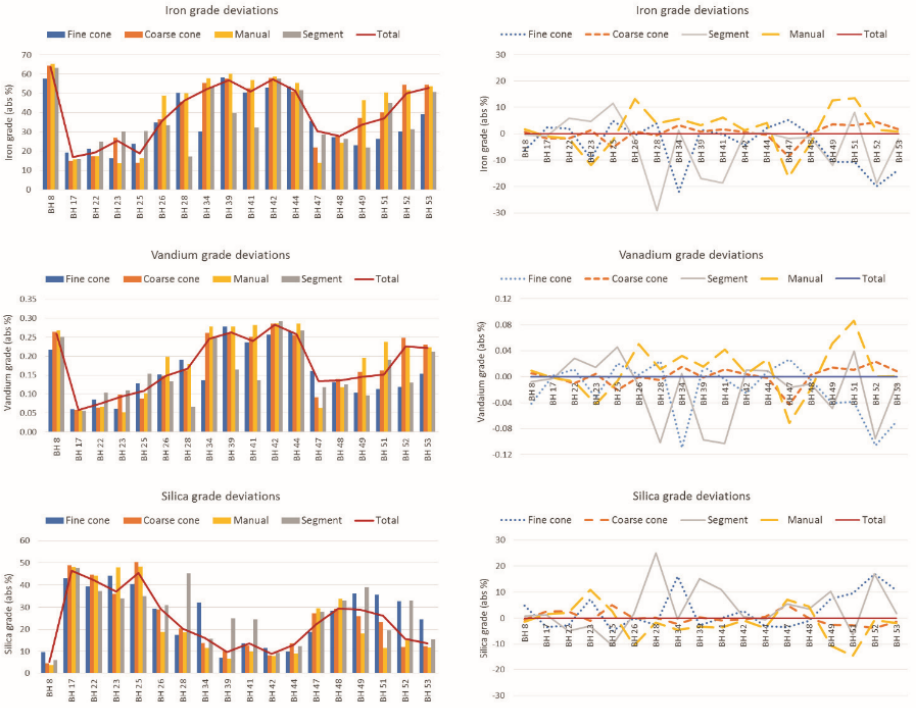

FIG 10 – Comparison of iron, silica and vanadium grade from 18 completely sampled blastholes to evaluate deviations for manual and segment samples in regards to the authoritative reference (the total weighted average from all collected material for each hole).

precise as the supposedly superior segment sampling. This somewhat surprising conclusion holds demonstrably for the investigated Leveäniemi ores in Svappavaara. If the results can be generalised to other ores in the LKAB's open pit mines and/or for still other iron mineralisations in general is a different matter.

Approximate relative standard deviations for the manual and segment sampling alternatives (both including sample preparation and analysis) are presented in Table 3. The values are calculated from the relative variograms created for each chemical element (but the evidence for silica is not used in the conclusions reached in this study). The nugget effect to sill ratio (indicating the magnitude of the operative TSE) is slightly lower for the manual method than for the segment method for iron grade, but less than half for vanadium. This numerical evidence might be interpreted to support the results for the manual method as being slightly more favourable for the specific Leveäniemi ores. However, there may be influencing factors related to the specifics of how the segment sampling method can perform when used with this specific drill rig (see discussion).

Particle size distribution results

The histogram in Figure 12 displays the critical size distribution evidence comparing all four BH samples: manual, segment, coarse cone, fines cone as well as the total weighted average result, ie the authoritative reference. The particle size relationships shown represent calculated averages over all blasthole analysis results, ie for each size class the results from all sampled blastholes have been used to create an average value for the four sample types. From this evidence it is clear that the manual sampling approach (which is strongly correlated with the coarse cone results) is consistently most similar to the authoritative reference results. It is notable, however, that even though the mass of the segment sample is on average 70 times greater than the manual sample, it deviates more from the weighted average total result than the manual sample at both ends of the grain size range. This might be the result of experienced sampling personnel being able to visually detect, and exclude, parts of the cone not originating from the drill hole but rather from filling material applied to the drilling area.

Both the segment data as well as the fines cone results display significantly deviating results across all sieve fractions investigated (the latter not surprisingly). Both these sample types deviate mostly at the grain size extremes, albeit in an anti-correlated fashion. While the segment samples underestimate the finest fractions with a concomitant overestimation of the coarsest fractions, the fine cone samples display the exact opposite behaviour.

The significant deviation in <63 µm size fraction, between total result and all other sample types, is unavoidable as long

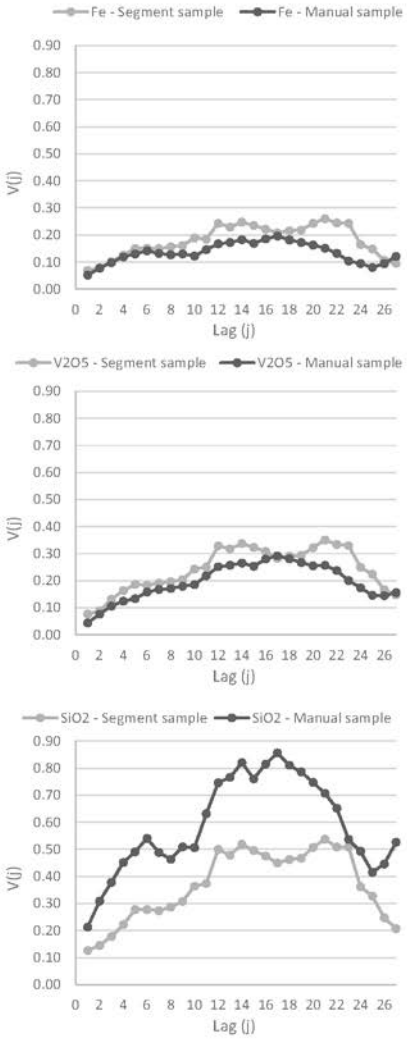

FIG 11 – Variograms for the important chemical variables iron, vanadium (and silica) comparing the manual and segment sampling methods.

as the drill rig separate the BH material in a fine and coarse cone and the fines cone is excluded from routine sampling. The obvious solution would be to extract the sample *before* the cyclone separation (see discussion section below). It is emphasised that only the manual and the segment sampling approaches are relevant for practical routine sampling of blastholes in open pit mines.

DISCUSSION

The results from both replication experiments and variographic characterisation surprisingly show that the manual sampling method generates equally precise and therefore reproducible samples as the segment samples. The results showed that the segment samples were more biased than has been previously shown in evaluations of sectorial cutters (Chieregati *et al*, 2015). One important difference is that the Atlas Copco D65 drill rig used at LKAB has the dust collector separating the coarse and fines cone (mandatory for occupational health and environmental reasons), which was not the case with the single-discharge drill rig used in the study by Chieregati *et al* (2015). In addition, occasionally the coarse discharge (where the sectorial cutter was placed during the variographic experiment) has a variable flow of material, both with regards to flux unit as well as with respect to ground position of the material stream from the outlet chute. When this happened it was impossible *a priori* to ensure that the sectorial cutter was placed precisely in the centre of the cone, which will affect the representativeness of the segment sampling method. If not placed consistently right at the centre of the outlet flux, a variable bias will result (smaller or larger as the off-axial placement would be). However, even though the discharge did exhibit variability in the material flux, through close communication with the drill rig operator, the sectorial cutter was in general able to be replaced to the centre of the pile after the first small amount material had been discharged from the drill hole. The variable flow of material might still have affected the representativity of the segment sample to a minor degree as the flow was not centred to the tip of the sectorial cutter at all instances during the drilling of one particular hole (see Figure 13). Although this occasional IDE infraction is unavoidable in practical BH sampling – the issue is if the variability originating from incorrect sampling errors is not larger than what will result in fit-for-purpose representativity corresponding to, say, a RSV below 10–15 per cent. This would be acceptable for the open pit ore/waste discrimination purpose in Svappavaara.

Chemical comparisons show that the manual and segment sampling methods are performing equally well with similar nugget effects (MPE), while the size distribution comparison show larger deviations for segment sampling at both ends of the size range. The size distribution correlations with the reference were good for the manual sampling approach with the notable exception of the <63 µm fraction, an obvious result of the exclusion of the fine cone when extracting the manual sample. The occasional coarse discharge alignment issues and the variations in the flux of material from the drill rig cannot

TABLE 3

Relative standard deviations for V(0), nugget effect and sill estimated from the relative variograms of the replication experiment performed in Leveäniemi open pit mine.

(Relative standard deviation)	V(0)		Sill		V(0)/Sill	
	Manual	Segment	Manual	Segment	Manual	Segment
Iron grade	3.2%	5.1%	18.6%	23.0%	17%	22%
Silica grade	13.3%	9.9%	79.7%	49.6%	17%	20%
Vanadium grade	2.2%	6.1%	27.5%	32.8%	8%	19%

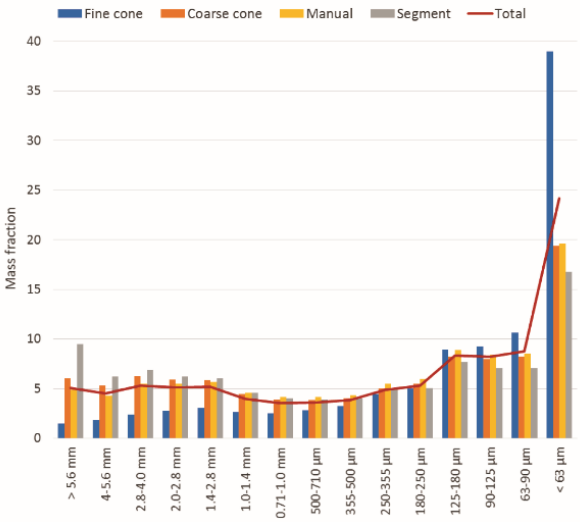

FIG 12 – Comparison of average particle size distributions for the four blasthole sample types. Thick line represents the weighted average authoritative reference calculated from all sample types (manual, segment, fines and coarse cone); see text for full explanation.

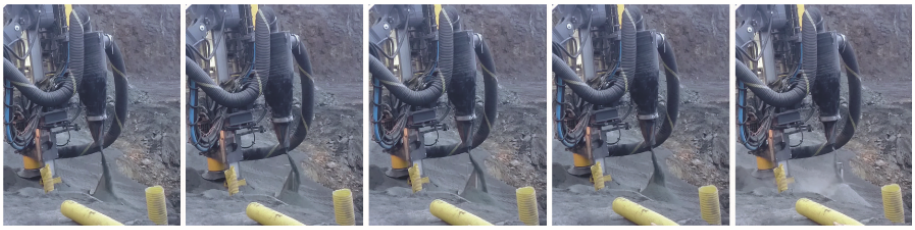

FIG 13 – Variability in the flux of blasthole coarse discharge from the D65 drill rig during experimental drilling.

be considered to be the sole reason for the partly surprising results regarding the relative performance of the alternative sampling approaches – as they only significantly affect a small fraction of the number of holes included in the RE and the variographic characterisations.

Even though the manual method shows best correlation to the weighted average total result, there are still some blastholes that show large variation between the different methods. These blastholes are the ones with the lowest iron grade and grade differences between fines and coarse BH cone, indicating that the manual sample method is fit-for-purpose when sampling higher grade iron ore which generally shows less heterogeneity. Lower grade ore is normally more heterogeneous and for these blastholes, larger deviations between both manual and segment sampling methods are evident.

To simply apply the manual method also to the fines cone is not a solution as the increment weighing error would be impossible to avoid in a practical set-up. Apart from this, the normal sampling situation where sampling is conducted after the drilling is finalised, often results in the fines cone being destroyed (run over by the drill rig), and therefore impossible to sample, as the discharge is located under the drill rig. The obvious solution to be able to collect a representative sample from all the BH material is therefore to sample *before* the dust collector that separates fine from coarse material. A single-discharge drill rig that generates one singular BH pile, for example the Pit Viper, would enable manual sampling from one limited lot of the complete BH material. However, the D65 rig with the dust collector actually also allows the possibility for attaching an automatic sampler before the dust collector to sample all the BH material in a fully TOS-compliant way (Pitard, 2004b).

It is worth noticing that BH sampling is currently applied to every second BH in the open pit, ie every 5 m. By comparison, the mine block used for short-term planning in Leveäniemi is 15 × 15 × 15 m, thus each block is characterised by an average of nine BH analytical results. This will improve precision for the block estimate by one-third (1/sqrt(9)) compared to the precision of the individual BH results, and thereby lowering the required quality demand for each individual analytical result. The blasthole analytical results are incorporated in to the original geological model (based on DD assays) to allow for more detailed short-term production planning. This

enables a continuous evaluation of the blasthole results as largely biased results can be identified when incorporated next to the DD results. The blasthole results are essential for the detailed short-term planning when classifying the quality of each ore block. The assays are used to delineate waste and ore contacts and are the basis for ore stockpiling and blending in to the primary crushing to ensure correct quality in the following refining and pelletising process.

CONCLUSIONS

This study has shown that the manual sampling approach in practice shows equal reproducibility as the sectorial segment sampling alternative, which runs contrary to most general expectations and scientific recommendations. As the iron grade for the mine blocks for short-term planning is based on nine BH sample results, the precision of the block estimate is believed to be *fit-for-purpose* in regards to the requirements for blending Leveäniemi ores into the primary crushers. The conclusion is based on extensive experience with the downstream LKAB processes. This study has shown that the general sampling variability of the currently used manual BH sampling approach is below ten per cent. The present findings apply to the specific ore types in Leveäniemi open pit mine and results cannot directly be generalised to lower grade iron ores with consistently larger heterogeneity.

For ore types where the iron grade deviates between fine and course fractions (ie BH numbers 49–53 in the variographic experiment), there are significant deviations between the manual sampling method and the authoritative reference, since the manual method does not sample the fines BH material separated by the dust collector. The only practical way to successfully eliminate this problem is to enable sampling before the dust collector and therefore sample all BH material at the same time. There would appear to be real possibilities to implement such an approach on the existing rig type, should so be desired/decided upon by LKAB's management.

The segment sampling method using a sectorial cutter is not by itself a representative alternative with the D65 drill rig as the sectorial cutter can only collect material form the course discharge. The method also has occasional issues with positioning the sectorial cutter correctly with respect to the outlet flux which will affect the representativity of this approach. In the experimental campaign it was found that radial segment sampling was affected by the potential alignment bias only to a minor degree, and this cannot in any way be claimed to be an explanation of the present findings.

The second part of the variographic experiment will address parallel RC drill sampling which will enable a critical comparison of the relative performances by variographics. These results are to be presented in an upcoming publication. Studies evaluating possible automatic sampling methods collecting samples before the dust collector on D65 rigs would certainly be of interest to improve BH sampling from this type of drill rig.

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Paper III

Optimal grade control sampling practice in open pit mining –
A full-scale Blast Hole vs Reverse Circulation variographic experiment

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RESEARCH ARTICLE

Optimal grade control sampling practice in open-pit mining – a full-scale blast hole versus reverse circulation variographic experiment

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ABSTRACT

Misclassification of ore grades results in lost revenues and the need for representative sampling procedures in open-pit mining is increasingly important in all mining industries. This study evaluated possible improvements in sampling representativity with the use of Reverse Circulation (RC) drill sampling compared to manual blast hole (BH) sampling in the Leveäniemi open-pit mine, northern Sweden. The results of the variographic experiment showed that sampling variability was lower for RC than for BH sampling. However, the total costs for RC drill sampling are significantly more than for manual BH sampling; other benefits may motivate the introduction of RC drilling. The main conclusion is that manual BH sampling can be fit-for-purpose in the studied open-pit mine. However, with so many mineral commodities and mining methods in use globally, there is no universal best practice for open-pit drill sampling and each case must be evaluated individually.

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Open-pit mining; iron ore; blast hole drilling; Reverse Circulation drilling; sampling; representative sampling; TOS; variography

Introduction

Owing to a marked slump of metal prices in the 10 years since 2008, the mining industry has struggled to increase productivity and simultaneously to lower production costs. An important aspect of the latter is to achieve successful short-term mine planning and thereby optimise resource utilisation by only processing ore above cut-off grade and to avoid loading any waste rock into primary crushers. Misclassification of ore types and grades leads to large revenue losses (Carrasco et al. 2004), and the need for representative sampling procedures is becoming increasingly important. As there is currently no universally agreed best practice sampling approach for open-pit mining, it is essential to adapt the sampling system to the specific ore types and to the local mining conditions. It is important to ensure that the drilling system used does not lead to hidden losses or missed profits due to unidentified sampling errors (Engström 2017).

The most common methods used for short-term planning in open-pit production are manual sampling of blast holes (BHs sampling) and Reverse Circulation (RC) drilling with an automated sampling system (RC sampling). During the last decade, there have been several publications that evaluated and compared the advantages and disadvantages of one or both methods (e.g. Pitard 2008; Abzalov et al. 2010; François-Bongarçon 2010; Chieregati et al. 2011; Gomes et al. 2011) but it has not been possible to draw comprehensive overall conclusions (Engström 2017).

According to Pitard (2008), Magri et al. (2011) and Chieregati et al. (2015), RC sampling gives more reliable results than BH sampling. It is argued that reduced sampling error effects justify the higher drilling costs for RC. On the other hand, it has also been shown that it is possible to achieve fit-for-purpose representativeness and to minimise loss of fines by conducting BH sampling in a well-controlled fashion using correct sampling procedures in full accordance with the Theory of Sampling (TOS) (François-Bongarçon 2010; Chieregati et al. 2011).

Previous publications aimed at comparing RC and BH sampling show inconsistent results depending on the mining methods, ore composition and the specific sampling methods used. It is notable that, out of 15 published comparisons, 7 claimed RC as representative and 5 used RC sampling as the reference against which BH sampling was evaluated (Engström 2017). This study aims to clarify whether BH sampling can match the same standard as RC in the case of open-pit iron ore mining.

Luossavaara-Kiirunavaara AB (publ.), known as LKAB, is an iron ore mining company in the north of Sweden. Its core business is producing high-quality iron ore pellets for blast furnaces and direct reduction steel making. Mine planning in LKAB open-pit mines is based on a resource block model with geological information derived from logging drill cores and chemical assays from diamond drilling (DD). The model is then completed in more detail using

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additional assay data from manual BH sampling. A previous evaluation of the current manual sampling approach and of an alternative, segment sampling method, was conducted through replication experiments and variographic analysis, both recommended by TOS. Results indicated that the manual sampling method is fit-for-purpose and equally representative in comparison with the segment method, for the chemical variables important for LKAB (Engström and Esbensen 2017).

Study objectives

The representativity of current BH sampling has been questioned by LKAB mine geologists. This initiated a comprehensive internal study of the accuracy and precision of the sampling method, which was completed in 2016 (Engström and Esbensen 2017). Even though the sampling scheme transgresses several TOS principles, causing incorrect delimitation error and incorrect extraction error (IEE) effects to some degree (Gy 1979; Pitard 2004, 2010), the results from the variographic experiment indicated that the manual sampling method is *fit-for-purpose* for the experimental transect studied.

The present study evaluated the alternative of using RC drill sampling instead of, or as a complement to, current manual BH sampling for possible improvements in sampling representativity. The objective of the study was to expand the drill sampling experiment conducted by Engström and Esbensen (2017) by adding another variographic experiment, specifically by drilling a complementing series with RC as twin holes to the previously sampled blast holes. This will enable both bias testing and comparative variographic analysis between the RC and the BH sampling methods. Apart from variographic analysis, the experimental data will also be assessed with conventional statistics and principal component analysis (PCA).

Material and methods

The present study was performed in the Leveäniemi open-pit mine, located in Svappavaara in northern Sweden. The regional geological plan, location plan and Leveäniemi geological map are presented in Figure 1. The Leveäniemi ore deposit of Kiruna type is hosted by metamorphosed rocks of volcanic and sedimentary origin, locally intruded by granite, pegmatite and diabase (Bergman et al. 2001). These meta-volcanic rocks, mainly trachy-andesitic in composition, are similar in age (1.88–1.91 Ga) and composition to the rocks of the Porphyry and Porphyrite groups in the Kiruna district some 40 km to the north-west (Eilu 2012). Massive magnetite ore dominates the Leveäniemi deposit, but alterations of haematite and martite are common in the central part of the deposit. In the peripheral zone, ore breccia occurs in zones up to 100 m thick, mostly with distinct contacts to the massive magnetite ore. The main gangue minerals in the deposit are apatite, calcite and amphibole (Bergman et al. 2001). The main ore type mined in the Leveäniemi open pit is the massive magnetite as well as magnetite veins and breccias in the trachyandesite units and biotite schist. The geological 3D model and resource block model in the Leveäniemi open pit are based on exploration DD carried out by the Geological Survey of Sweden in 1957–1962 (Bergman et al. 2001) and by LKAB in the 2000s. Manual BH sampling is currently used for short-term mine planning and grade control in all LKAB open-pit mines. Production planning is based on a 3D block model, in which the chemical assays from exploration drilling and manual BH sampling are combined.

Blast holes in the Leveäniemi open pit are drilled using an Atlas Copco D65 BH drill rig equipped with a cyclone dust collector that separate coarse and fine material in two BH cones (for occupational health and environmental reasons). The major part of the drill cuttings is ejected at the front of the rig, while a smaller fraction of fines is ejected under the rear end of the rig. During the current manual sampling protocol, every other BH is sampled manually and the sample extraction is applied to the coarse BH cone only. The manual sampling procedure starts with cutting away a sectorial part of the BH cone. The sample is then collected as a vertical slice of uniform thickness in the exposed axial centre of the cone. The sampling protocol aims at collecting an equal amount of material from each cone level. The sample extraction is always carried out from the bottom to the top of the cone to avoid spillage. It can be noted that the manual BH samples are extracted after all drilling has been finished in order to minimise the health risk for sampling staff. All primary samples are then sent to the laboratory for chemical analysis. The deliberate exclusion of the smaller BH cone with finer material is a clear violation of the Fundamental Sampling Principle (FSP) and the current manual BH sampling method will likely induce IEE effects (DS 2013).

BH drill sampling campaign

BH drill sampling was conducted using an Atlas Copco D65 rig, which is normally used by LKAB for production drilling and manual sampling in open-pit mines (Figure 2). During the BH campaign, a total of 53 blast holes were drilled. The samples were extracted using the current manual sampling method as well as an alternative full segment approach, which uses a sample frame collecting a complete sectorial part of the BH cone (Figure 3). Both the manual and segment sampling methods were only applied to the coarse BH cone as this is the only feasible option in day-to-day

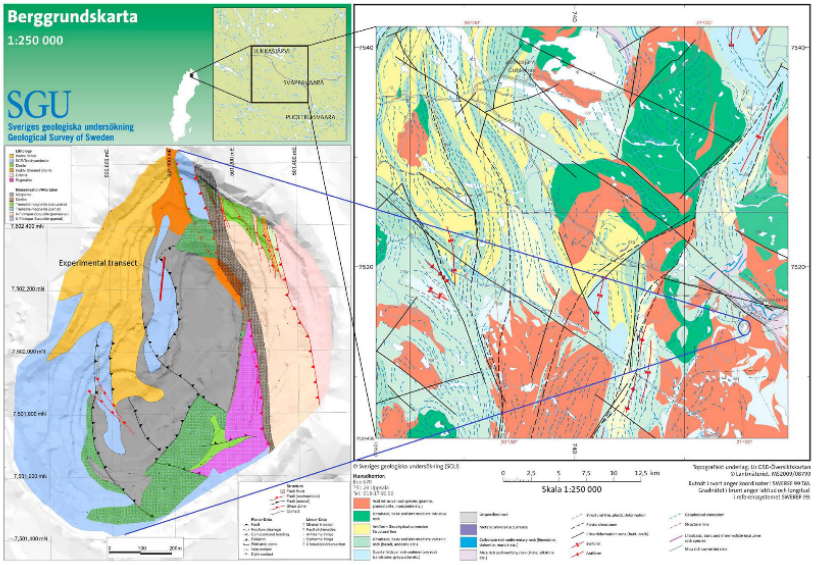

Figure 1. Regional geological map (top right) with reference to the location in Svappavaara in northern Sweden. Local geological plan for Leveäniemi open-pit mine (bottom left). The thick line on the Leveäniemi geological plan marks the experimental transect in the current study.

production. The fines discharge located underneath the drill rig is not accessible during routine production BH drill sampling.

During the present experiment, all of the remaining BH drill cuttings, from both the fines and coarse BH cones, were collected from 18 of the 53 blast holes. The full cone samples were weighed and split representatively in the field, using TOS-correct riffle splitting (Figure 3). This resulted in 18 blast holes with four alternative partial sample types: a manual BH sample (current routine protocol), a full segment BH sample (supposedly an improved sampling method), a fines cone sample and a sample consisting of all the remaining coarse cone material. This allowed aggregation of

Figure 2. Experimental drilling with a canvas located under the fines cone discharge (rear end) and a canvas and sectorial sample frame located under the coarse cone discharge under the front discharge cyclone of the Atlas Copco D65 drill rig.

Figure 3. Full-scale BH sampling experiment. Top left: Segment sampling frame placed in coarse BH cone. Top right: Manual and segment BH sample extracted from coarse BH cone. Bottom: Extensive in-field splitting and weighing to reduce the full coarse BH cone and fines BH cone samples to laboratory size. All field splitting was fully TOS-compliant.

an authoritative reference result for each BH, which was calculated as the weighted average from the assays of the four partial samples. The weighted average coefficients were based on each partial sample mass. The scope, method and results of the separate BH variographic experiment are presented in detail in Engström and Esbensen (2017).

RC drill sampling campaign

The RC drilling campaign was carried out using an Atlas Copco Explorac 100 drill rig (Figure 4). The drill rig was equipped with Atlas Copco Secoroc, down-hole drill bits, during the experimental campaign. The Explorac uses compressed air to transport drill cuttings from the hole to the cyclone which separates the air from the cuttings and passes these through a tiered riffle splitter to reduce sample size. Field RC samples were collected for every 1 m depth, i.e. 20 partial depth samples per drill hole, and brought directly to the laboratory. Here, each sample was split to 600 g (using a TOS-correct riffle splitter) and then combined into four 5 m depth interval composite samples per RC drill hole, for XRF analysis.

The RC drill holes were deployed along a parallel profile with an average distance of 1.5 m from the earlier drilled BH profile. The spacing between RC drill holes was 2.5 m along the strike (Figure 4). Both BH and RC holes were 20 m deep with equal dip. The sampling experiment was conducted along a transect of significantly varying Fe grades. This secured that the 55 BH and parallel RC drill holes would allow a test of the validity of the two alternative sampling methods to be compared. The set of parallel BH and RC sampling campaigns enabled three variographic characterisations, one for each principal sampling method. Bias testing was also applied, comparing RC sampling to manual and segment BH sampling as well as to the authoritative full BH cone reference sample results (Engström and Esbensen 2017).

Figure 4. Explorac 100 RC drill rig in action during the twin hole drill sampling experiment (winter in northern Sweden). The tubes mark the Blast Holes transect whereas the exposed rocks mark the twin RC holes.

TOS and the semi-variogram

The process of taking a small part of a lot and receiving an analytical result thereof is trivial, but to collect a representative sample from the same lot and receive a valid and reliable analytical result is more challenging. The TOS was first developed by Pierre Gy in the 1950s. It has since reached the level of a complete scientific theory, covering all aspects of sampling particulate materials. The main purpose of sampling is to reduce the mass of the lot to be assessed to the size of the analytical aliquot in a fully representative manner. In order to achieve representative analytical results, a comprehensive understanding of the principles and practices within TOS is essential (Gy 1979; Pitard 2004, 2009; DS 2013). The FSP states that all parts of the lot must have equal probability to be selected for a sample (Pitard 1993; DS 2013). This means that any sampling situation needs to be developed so that every particle in the lot has an equal opportunity to end up in the final analytical aliquot. To achieve this, the primary sampling as well as all subsequent sampling and preparation stages need to be carried out in strict accordance with TOS principles for representativity. A comprehensive summary of TOS is presented in several publications (e.g. Gy 1979; Pitard 1993; DS 2013).

Variography is a powerful tool for characterisation of time- or space-serial data sets. The semi-variogram can be used to quantify different types of heterogeneity fluctuations present in the data (Pitard 1993). Variographics allow separation of variability stemming from the sampling system itself (correct and incorrect sampling errors) from the true variability in the process, or along a profile, as evidenced by the analytical results from the sampled material. The calculation of the point values in the semi-variogram is presented in Equation (1), and the main parameters of the variogram are described in Table 1. Q is the total number of measurements, equidistantly distributed in time or space. The dimensionless distance between the measurements is called the *lag: (j)*. The *nugget effect*, termed $V(0)$, of the variogram is defined by back-

Table 1. Important parameters for characterisation of the relative variogram (Minnitt and Pitard 2008; Esbensen and Julius 2009; Esbensen and Paasch-Mortensen 2010; Minkkinen 2013; Minnitt and Esbensen 2017).

Notation	Definition	Description
$V(0)$ – Nugget effect	Extrapolated intersect of y-axis	Short range random variability stemming from the total measurement system, including sampling, sub-sampling, sample preparation and analytical measurement. The nugget effect is the summation of both correct (CSE) and incorrect sampling errors (ISE), as well as the total analytical error (TAE)
$V(1)$	Value at $V(1)$	The total variability representing the time period between two samples, including process and sampling variability
V(Process)	$V(1) - V(0)$	Process variability for the time period between two samples, not including the total measurement system variability. The relationship between V(process) and $V(0)$ is a good indication of the possibility of controlling the process with current sampling interval
Sill	Ceiling level of the variogram	Describes the highest variability, or the global heterogeneity, in the data series. Technically the sill is the average of all variogram point values
Range	Where the variogram reaches the sill	For process variograms the range can be directly translated into a time interval where paired samples show auto-correlation. The range is where an increasing variogram has flattened out

extrapolation to the y-axis intersect of the variogram. This value is an important parameter as it improves an estimation of the total error variance of the measurement system, including primary sampling, sample preparation and analysis (Esbensen and Paasch-Mortensen 2010; Minkkinen 2013; Minnitt and Esbensen 2017). The nugget effect can be subtracted from the total variability estimated by the *sill* in order to gain insight into the true ore heterogeneity along the profile.

The method of variographic characterisation of time or space sequential data, including the calculation of the relative semi-variograms, has been described in numerous reference publications (e.g. Pitard 1993; Gy 1999; Minnitt and Pitard 2008; Esbensen and Paasch-Mortensen 2010; Minkkinen 2013; Minnitt and Esbensen 2017). Samples can either be characterised by their analytical concentration a_q, or by their relative heterogeneity contributions: h_q (Equation (2)), resulting in a shape-similar relative semi-variogram (Pitard 1993; Gy 1999; Minnitt and Pitard 2008; Esbensen and Paasch-Mortensen 2010; Minnitt and Esbensen 2017). For comparison between variograms from different profiles, it is easier to use the relative variogram.

$$v(j) = \frac{1}{2(Q-j)}\sum_{q=1}^{Q-j}(h_{q+j} - h_q)^2 = \frac{1}{2(Q-j)a_L^2}\sum_{q=1}^{Q-j}(a_{q+j} - a_q)^2, \quad (1)$$

$$j = 1, 2, \ldots, \frac{Q}{2}$$

$$h_q = \left(\frac{a_q - a_L}{a_L}\right)\frac{M_{s_q}}{\overline{M_s}} \quad (2)$$

$v(j)$: variogram point estimations Q: total number of measurements used for variographic characterisation j: lag (distance between samples in time or space) h_q: relative heterogeneity contribution for the sample q a_L: mean analytical concentration for the lot a_q: analytical concentration for sample q M_s: sample size

Data set information relationships

From a geological and mining point of view, RC and the BH data are supposed to carry the same information, disregarding or compensating for the total sampling error effects and the analytical uncertainty involved. In this study, the analytical uncertainty was equal for both data sets as all samples were subjected to identical protocols for splitting, crushing and grinding, and were analysed in the same laboratory. This means that only the different drilling and sampling approaches affected observable differences. For comparison between RC and BH data, there was an additional error source stemming from the ore variability between twin holes. This effect from the *in situ heterogeneity* of the ore was manifested on a scale of 1.5 m, the distance between the RC and BH drill profiles (Figure 4).

Multivariate data analysis

The raw data from observations and experiments seldom hold useful information in themselves until appropriate data analysis has been applied. The possibility to extract useful information is dependent upon whether the data actually contain relevant information relative to the purpose of the experiments. To be able to retrieve information from the recorded data, proper definition of the analytical problem and well-planned experiments are essential. It is more important to measure meaningful variables than to simply take a large number of measurements (Esbensen 2009).

Describing material properties in empirical sciences and process industries, such as mining and mineral processing, is often a multivariate task. PCA uses orthogonal transformation (projections) of the original data into a set of uncorrelated linear combinations of the original variables, called principal components. The first few components describe the largest variances in the original data (in descending order) and the later components are often considered to be non-significant and can therefore be eliminated, allowing the underlying data structure to be modelled in a simplified manner but retaining all the important information.

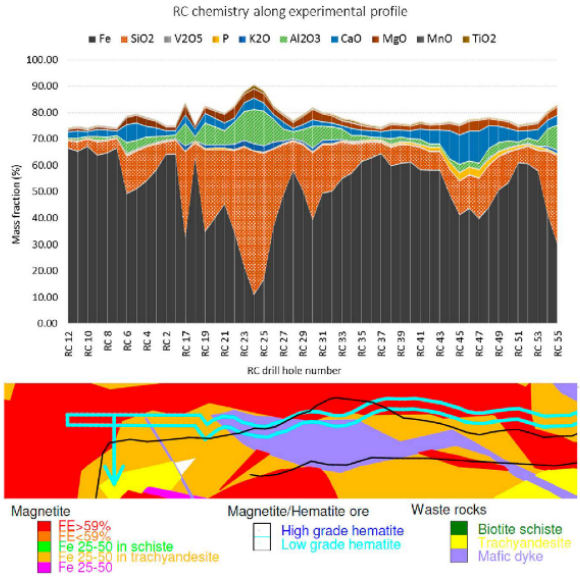

Figure 5. Upper pain: Chemical composition of the RC samples along the experimental profile, documenting a highly variable ore composition. The remaining percentages to reach 100% are due to oxygen bound to iron oxides. The XRF analysis does not indicate the grade of iron oxide but rather the grade of free iron. Lower pain: Corresponding area from the block model with the experimental transect marked. Close correspondence between high and low iron grade areas can be seen between the model and RC sampling results.

PCA is the most widely used multivariate method for finding hidden data structures, to reduce the number of original variables and express the data through a few principal components. These can be used for direct interpretation, input for linear regression, linear discriminant analysis and artificial neural networks for example (Esbensen and Geladi 2009). PCA can discriminate between systematic information and noise in the data, thereby minimising the number of components needed to describe the original data. PCA can also detect outliers and disclose patterns and correlation between variables and samples (Esbensen and Geladi 2009). The full theory and basic mathematical calculations for PCA are well described in the literature (e.g. Brereton 2003; Esbensen 2009; Esbensen and Geladi 2009; Bro and Smilde 2014) and will not be further described here.

The analytical results from the current experimental campaigns are indeed multivariate as 10 chemical elements are analysed for each sample. To achieve a comprehensive understanding of the complete variability and correlation in the data from BH and RC experiments, respectively, as well as the combination of the two, PCA has been applied to both types of data. This makes it possible to see overall data structures and also to determine which elements contribute most to the systematic variability in, and between, the two data sets.

Results

RC drilling resulted in a total of 50 drill holes. Unfortunately, the planned hole number RC 20 in the centre of the profile could not be drilled and sampled due to extraordinary ground level variations that made it impossible for the rig to access the planned position. An approximate assay result for this hole has been estimated as the average of the two adjacent holes simply to allow for proper variographic characterisation in this study. This did not result in any perceptible adverse data modelling bias. For the statistical analysis, BH and RC results from the 50 parallel twin holes were used. The remaining BH results were excluded from the analysis as no corresponding RC results were available. The RC assay results exhibited similar ore variations to those of the BH drill sampling from the experimental profile. All results correlated well to the block model for the specific area, i.e. areas of high and low iron grade in the 3D model conformed well to the analytical results from the drill sampling campaigns. The full chemical variability in the ore as revealed by

RC results, together with the corresponding area of the Leveäniemi mine block model, is presented in Figure 5.

PCA evaluation of drill sampling methods

A multivariate PCA approach is eminently useful for making an overall comparison between the two drill sampling methods. The data were auto-scaled using standardisation before model calculation. Even though all data are measured in the same unit, given in mass percentage, the grade differences between the elements are large. Scaling was therefore needed to allow the relative variability for all elements to have an equal effect on the multivariate models (Brereton 2003; Esbensen 2009; Esbensen and Geladi 2009; Bro and Smilde 2014).

The scores and residuals plots for the PCA are presented in Figure 6. The samples originating from the two drill sampling methods are annotated with filled circles for BH samples and open circles for RC samples. The labels in the score plot indicate the hole number for each drill hole, where every BH-RC twin hole is annotated with the same running number. The residuals plot for the PCA model shows a clear *outlier* (sample BH 47; Figure 6). Looking more closely at this sample, it was found that it also deviated from both the parallel segment sample and authoritative reference. With this information, the sample was considered an outlier. For optimised data analysis, the results from BH 47 and RC 47 were therefore removed from the data set and a second PCA model was created (Figure 7).

The result from the outlier-screened PCA analysis shows that most of the variability in the data set can be explained by three components. Component 1 is a reflection of the important and strongly correlated variables Fe and Si grade as well as V and several other minor elements in the samples. Fe and Si are strongly negatively correlated to each other, with a correlation coefficient of $r^2 = 0.94$. PC1 accounts for approximately 56% of the total data set variance (Figure 7). PC2 adds 30% to the modelled variance and is mainly driven by phosphorous and calcium. There are only a few samples (44–46 and 48) with high concentrations of apatite, calcite and actinolite (amphibole), which are the source for this orthogonal variability. It can be noted that these samples show a weaker correlation between Fe and Si. The correlation coefficient between Fe and Si grade increases from 0.94 to 0.99 if these five samples are removed before univariate regression. PC3 describes an additional 8.5% of variability, almost solely described by titanium (ilmenite or titanite). Only a few samples (23–25), likely from a mafic dyke, are responsible for this variability contribution (Figure 7).

This study has emphasised the major magnetite variability seen in a large portion of the analysed samples. Furthermore, it is useful to know that the apatite, calcite, actinolite, ilmenite and titanite modality changes can be captured and thus decomposed by the second and third principal components. The optimised PCA data model illustrates that the important Fe/V↔Si variability is dominant along the profile selected. As such, the comparison results below can be said to have a very high relevance for this type of iron ore.

The score plot shows that the difference between the sampling methods is small for many of the twin holes located in the upper left quadrant, whereas the discrepancy is larger for other twin holes. The loading plot shows that the twin holes with smaller differences relate to areas with high Fe grade in the open pit. A notable observation is that the largest discrepancies between BH and RC samples can be seen for the samples shown to span components 2 and 3 in the PCA analysis. These samples show lower Fe grade and higher P, Ca and Ti grade compared to the majority of the samples. The data indicate that these drill holes intercept areas of trachyandesite that is high in apatite, calcite and actinolite (amphibole),

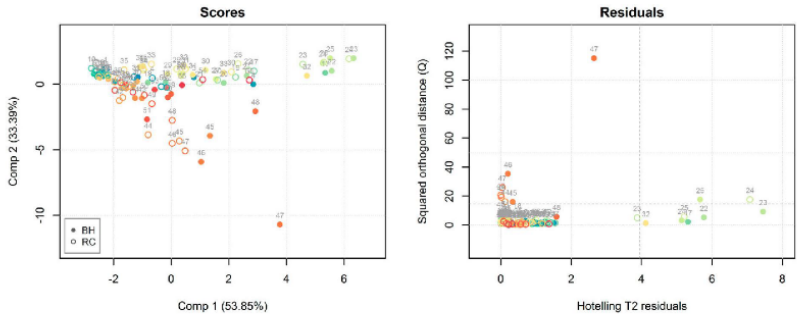

Figure 6. Scores and residuals plot for PCA of the complete data set from both BH and RC drilling. Filled circles represent BH samples and open circles are RC samples. Note sample number 47, inferred to be an outlier.

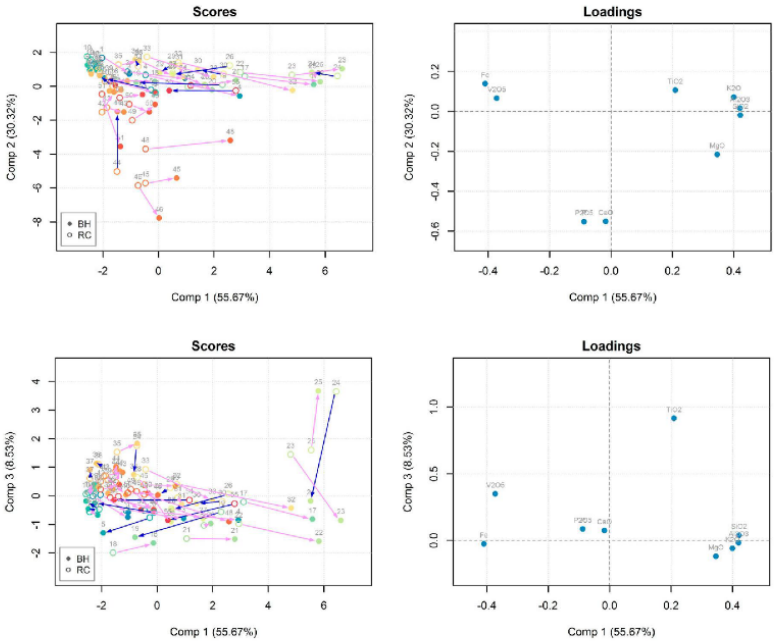

Figure 7. Scores and loadings plots for components 1 vs. 2 and 1 vs. 3 for the PCA with the twin BH47 and RC47, outliers removed. Filled circles represent BH samples and open circles represent RC samples. The arrows connect the twin holes to indicate the pair wise differences.

and mafic dykes that are high in ilmenite and titanite. This conclusion is supported by the location of the specific drill holes in the geological block model (Figure 5).

Univariate comparison of RC and BH drill sampling methods

For the critical elements Fe and Si, univariate comparisons between the manual BH and RC drill sampling are presented in Figure 8. The univariate regressions indicate, similar to the PCA analysis, that the correlation between results from the alternative methods is weak. This is most evident for samples with low Fe and high Si grade, i.e. the samples with deviating mineral composition spanning component 2 and 3 in the PCA analysis.

The manual and segment BH sample data as well as the RC results are compared to the full BH cone reference data (Figure 9). These results indicate that the RC sample types correlate equally well to the authoritative reference as the manual BH method for the major part of the twin hole profile. However, the RC results tend to overstate Fe grade and understate Si grade, compared to the reference. This result can possibly be explained by loss of lighter silica particles during RC drill sampling.

The largest differences between the RC and the reference samples were found in drill holes 17, 22 and 47–52. These correlate to the same drill holes that were the main drivers for principal components 2 and 3 in the PCA analysis, where higher concentrations of apatite, calcite, actinolite (amphibole), ilmenite and titanite are affecting the samples. These results support the conclusions from Engström and Esbensen (2017) that the ore was more heterogeneous in these parts of the experimental profile.

Variographic analysis

Variographic analysis was applied to the twin RC drill sampling profile as well as to the manual and the segment BH sample profiles. The variographic experiment resulted in a total of 50 twin holes for which both RC and BH drilling produced acceptable samples. For the one missing RC drill hole (RC 20), the average assay results of the two adjacent RC drill holes was used; this does not influence the

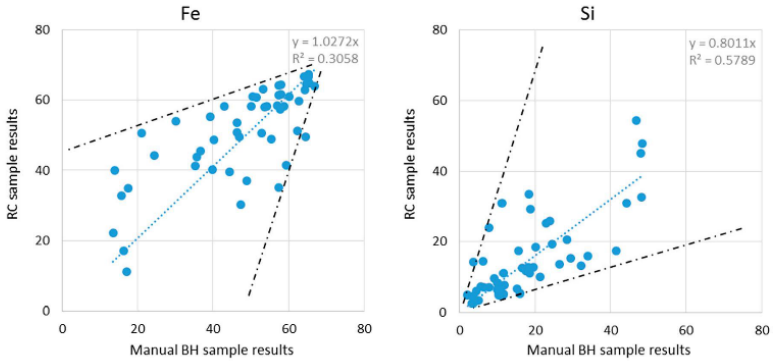

Figure 8. Correlations between parallel BH and RC samples for Fe and Si grade.

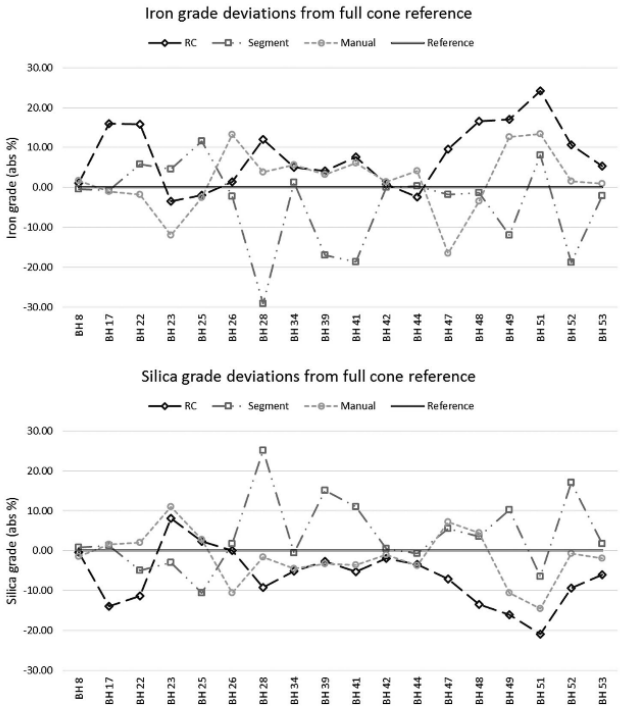

Figure 9. Comparison of Fe and Si grade between RC samples, manual BH samples and segment BH samples in relation to the authoritative BH reference, which would be the ideal field sample, although unrealistic for regular short-term mine planning.

variogram calculations unduly. Equally, for the BH outlier (BH 47), an average of the two adjacent assay results was used.

The sampling variability of the manual and segment BH samples has previously been demonstrated to be fit-for-purpose for the intended use (Engström and

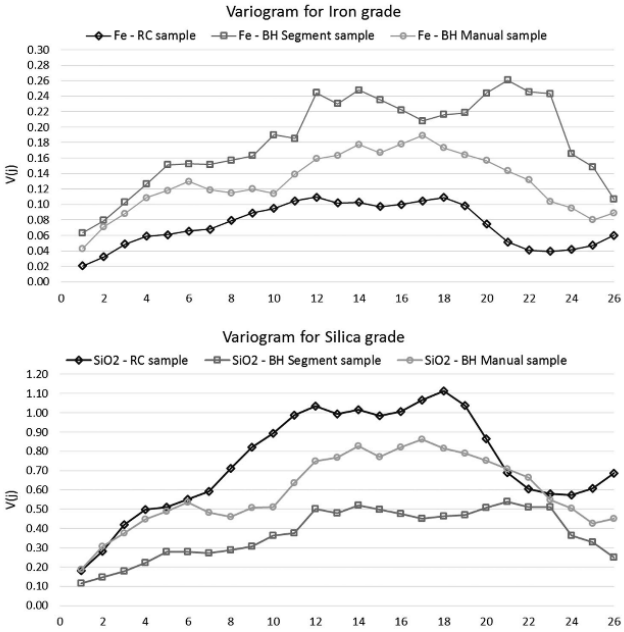

Figure 10. Relative semi-variograms for Fe and Si grade for the three drill sampling methods.

Esbensen 2017). The RC approach evaluated in this study was compared to both previously studied BH sampling methods to evaluate the performance in regards to sampling variability. Relative semi-variograms for the three sampling methods for Fe and Si grade are presented in Figure 10.

For Fe grade, the nugget effect, as well as the sill, is lower for RC drill sampling than for the two BH sampling methods. The range for all variograms is similar, around 5–6 lags, or 12–15 m, along the profile. Si grades show similar nugget effects for all sampling methods, whereas the sill is higher for RC and manual BH sampling. The range for the Si grade variograms is also reached around 6 lags, or 15 m, due to the correlation between Fe and Si grade along the profile. The large sill differences between the relative variograms for Fe and Si grade are explained by the average Fe grade being three times higher than the Si grade, whereas the overall variability of Si is equal to that of Fe. The different sill levels of the three variograms are a direct reflection of the magnitude of the total variance displayed along each profile.

It is of interest to compare the nugget effect to the sill level in any variogram. The nugget-to-sill ratio is an estimation of the percentage of total variability seen in the assay results that stems from the total measurement system (primary sampling/splitting/analysis). By evaluating this ratio it is possible to assess the quality of any sampling system. A recent introduction to this feature has been presented by Esbensen and Romañach (2015). In the present study, the nugget-to-sill ratio for Fe and Si grade is lower for RC sampling than the manual BH sampling. The ratio for the BH segment sampling is in turn higher than for the manual BH sampling method. This indicates that the RC sampling method exhibits less sampling variability than the two BH sampling methods.

Discussion

The results from both univariate and multivariate analyses indicate that the correlation between the twin holes for manual BH sampling and RC sampling is weak. This is especially the case for lower Fe grades and mineralisation with high proportions of gangue minerals (apatite, calcite, actinolite, titanite and ilmenite) responsible for PC2 and PC3 in the PCA analysis. One reason is that the ore heterogeneity for average to low Fe grade areas increases the sampling variability significantly. The comparison showed similar differences between manual BH samples and RC samples in regard to the BH authoritative reference. This indicates that the sampling error effects for manual BH samples are equal to the RC sampling and the between

twin hole heterogeneity combined. It has previously been indicated that twin holes less than 5 m apart are necessary in order to produce a valid basis for comparison of sampling methods (Abzalov 2009). The present study, however, indicates that even 1.5 m can result in significantly large ore variations between twin holes. The weak correlation between RC and BH sampling in this study is partially due to the large degree of ore heterogeneity at this scale which is increasing the *in situ* sampling variability. The BH results are, furthermore, not directly comparable to the corresponding twin hole RC results due to significant sampling variability. This is evidence that the specific ore body characteristics must be taken into account when comparing sampling techniques.

The variographic results show that implementing additional RC drill sampling could improve the sampling variability for short-term grade control in the Leveäniemi open-pit mine. In addition, RC sampling delivers 1 m interval samples from the complete depth of the hole, enabling any number of highly resolved assay results from each drill hole. In heterogeneous parts of the ore body, this can be valuable in ensuring more detailed control of material classified as ore or waste, respectively. As BH sampling only generates one assay result from each 20 m BH, the whole bench height is classified according to an average of the ore grades present at various depths of the bench. From the viewpoint of the mine geologists and mine planners, this could be more important for effective short-term production planning, than the increase in sampling precision that RC sampling could achieve.

The costs for RC drilling and subsequent sample handling exceed the current costs for manual BH sampling of production drill holes. Even if the RC drill holes can be used for subsequent blasting, and more extensive and long-term drill campaigns reduce the price per drill hole compared to the small campaign in the current study, the RC drilling costs are still higher than for BH drill sampling. Furthermore, RC drilling requires a separate drill rig. The extended amount of sample handling would also increase costs and needed resources, both in regards to the collection of relevant field samples and for the increased amount of splitting, crushing and assaying in the laboratory. Compared to the current manual BH sampling method where one primary sample of approximately 1 kg is collected from each BH for the laboratory, RC sampling would generate one primary sample, of approximately 3–5 kg, for each metre drilled. This means a considerable increase in the capacity needed in the laboratory in terms of both equipment and manpower. The increased cost needs to be assessed against possible economic benefits following improved ore and waste classification reliability and increased precision of sampling based on RC drilling, which was not incorporated in this study.

Conclusions

The conclusion from the variographic characterisation in this study is that RC sampling is able to generate lower sampling variability compared to the current manual BH sampling method. However, the manual method can still be characterised as fit-for-purpose for short-term mine planning in the Leveäniemi open-pit mine (Engström and Esbensen 2017). The possibility of dividing the 15–20 m benches into smaller vertical strata when using RC drilling, could be valuable for improved geological modelling in areas of the mine where the ore grade variations are high. With the present mining strategy, however, the value gain from this does not appear to be able to offset the increase in both drilling costs and resources needed for sample handling in the field and in the laboratory.

The final conclusion from this study is that RC drill sampling is not sufficiently superior to the manual BH sampling method to justify the significantly increased cost for drilling and sample handling in the studied mine. Previous publications (e.g. Magri and Ortiz 2000; Crawford et al. 2005; Pitard 2008) have concluded the opposite with regard to the superiority of RC drill sampling. This indicates that both the drilling and sampling methods need to be carefully evaluated for each case and validated with respect to the specific ore body characteristics and local mining situations.

It is not possible to draw general conclusions from the results from this study as to one, universal best practice sampling approach. Instead, we have to be content with a more fully developed understanding of the specific heterogeneity characteristics in the studied open-pit mine. Based on the above, the general recommendation is to encourage more extensive use of well-planned pilot variographic sampling experiments, along a profile with relevant ore heterogeneity, before opening up new areas in open-pit mining. There are significant advantages in the use of variographic profiling, which will allow mining geologists and production engineers to evaluate the specific total measurement system performance in direct relation to the full ore variability observed along the profile.

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Disclosure statement

The first author is a full time employee of LKAB, where the present study was conducted. The study is also one part of a LKAB funded industrial PhD project, conducted by the first author and supervised by the co-author.

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Paper IV

Experimental evaluation of surface water sampling variability for environmental monitoring in iron ore operations using concepts from the theory of sampling (TOS)

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Experimental evaluation of surface water sampling variability for environmental monitoring in iron ore operations

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Abstract

Environmental self-monitoring is a government requirement for Swedish process industries. This includes sampling and analysis of recipient water that might be adversely affected by emissions from the process. The requirements for accredited analytical methods, with well-defined measurement uncertainties are strict, but estimations of the attendant sampling variability are seldom required, presented or evaluated in environmental surface water sampling. The goal of this study was to perform an initial evaluation of the measurement variability for surface water sampling within the self-monitoring program for a large mining company in northern Sweden. The results indicate that the method for evaluation of sampling and measurement variability itself affects the results obtained. Therefore, the evaluation scope must be clearly defined in advance, so that the most appropriate approach, resulting in a realistic quantification of total variability, can be selected. This study shows that duplicate sampling experiments result in significantly larger sampling variability estimates when accounting for ambiguities in the sampling protocol, than similar experiments under repeatability conditions. This is due to large temporal variations in stream flux and analyte concentrations in the evaluated sampling targets. In order to allow valid evaluation of the total sampling and measurement system variability, the ambiguities in different sampling protocols must be fully described and considered when designing empirical evaluation experiments. To reduce unnecessary sampling variability, and to be able to cover the significant temporal changes in stream flux and analyte concentration appropriately, an automated sampler using volume-proportional sampling to collect increments for composite samples, is recommended.

Keywords: sampling; recipient water; duplicate sampling; replication experiment; Theory of Sampling; TOS

1. Introduction

The Theory of Sampling (TOS) is a complete theory covering all aspects of sampling particulate material. Pierre Gy started the development of TOS in the 1950's and all major parts of the theory of sampling is presented in e.g. Gy (1979) and Esbensen (2016). Even though TOS was originally developed for sampling of particulate materials, all main principles related to sampling errors and heterogeneity can be applied to practically any form of material sampling, including surface water (Gy 2004). Environmental self-monitoring is in most countries mandatory for industries that might affect the environment adversely. The company is responsible to control the impact on the environment through self-monitoring which should be internally developed based on the operations at hand. Even though some countries require mandatory education of water sampling staff, the sampling methods used before analysis are often *not* in compliance with TOS. In surface water sampling, as in any other sampling situation, the heterogeneity of the target lot will influence on the validity of sampled water/material. It is essential to consider and counteract the lot heterogeneity in any sampling protocol. This is done by collecting equi-probabilistic samples where all parts of the lot must have an equal probability to end up in the sample (Pitard 1993; Gy 1998, 2004). Only by following this Fundamental Sampling Principle (FSP), and all complementary principles laid out by TOS, can a documented representative sample ever be collected (Esbensen and Wagner 2014).

Analytical results will always be accompanied by an uncertainty arising from remaining, or insufficiently reduced sampling errors, in addition to the analytical uncertainties. Sampling errors are divided into two categories: *correct* sampling errors (CSE) and the *incorrect* sampling errors (ISE). The correct sampling errors include the fundamental sampling error (FSE) and the grouping and segregation error (GSE), which are always present in all sampling situations, but can be *minimized* using correct sampling protocols. The incorrect sampling errors: incorrect delimitation error (IDE) and incorrect extraction error (IEE) relate to the geometrical delineation and physical collection of the sample (increment), while the incorrect preparation error (IPE) relate to the post-sampling processes itself including sample preparation and possible contamination during the activities carried out on the sample between primary sampling, sub-sampling (mass reduction) and chemical analysis (Esbensen and Paasch-Mortensen 2010). The ISE must be eliminated in order to be able to guarantee a bias-free sampling process (DS 3077 2013).

Even though it is claimed to be widely agreed that the measurement process starts when the primary sample is collected from the lot, the analytical uncertainty of chemical measurements is traditionally the main focus of the measurement uncertainty ($MU_{analysis}$) of reported analytical results (Ramsey et al. 2011). However, the complete measurement uncertainty $MU_{sampling+analysis}$ including the variability stemming from sampling (primary and sub-sampling), is still seldom presented or even evaluated in environmental surface water sampling (Esbensen and Wagner 2016).

Several recent publications have acknowledged this lack of focus on sampling variability in environmental monitoring, presenting examples of experimental methods used for estimating both sampling and analytical uncertainties in various environmental applications (Botta et al. 2012; Guigues et al. 2016; Ramsey 1998, 2011; Rode and Suhr 2007). The increased scientific understanding and focus on the importance of sampling variability in environmental control programs can be expected to influence the governmental control of mining operations and other industries affecting the environment by air and water emissions. This prospect is an incentive for the industry to act proactively by including control of sampling variability in the mandatory self-monitoring programmes related to environmental analysis. It has since long been mandatory to evaluate and document the analytical measurement uncertainty, including any contribution from analytical bias within the laboratory, together with any accredited analytical result. However, to account for the uncertainty stemming from primary sampling in the field, and secondary sampling operations in the laboratory, is not generally expected in environmental monitoring. This paper is a first foray pointing out the consequences that will result if this status quo continues unchanged.

1.1. Environmental monitoring and governmental regulations in Sweden

Luossavaara-Kiirunavaara limited company (LKAB) is a state-owned mining company in the north of Sweden. The core business is producing high quality iron ore pellets for blast furnace and direct reduction steel making. According to governmental regulations in Sweden, the operator must provide the knowledge and evidence of the environmental impact attributed to the operation. This is done through self-monitoring which is a continuous process, usually containing sampling and analysis, studies and investigations. If the environmental permit is associated with specific terms, these are to be controlled by the self-monitoring and results are reported to the authorities. How to perform, follow up, report and document the self-monitoring is described in a control program, normally including sampling protocols. These protocols also describe sampling targets and frequencies, as well as the necessary requirements for analytical parameters and analytical accreditation. Monitoring and control of the analytical uncertainty for each accredited analysis is closely controlled by accreditation officials. However, the sampling variability coupled with the specific surface water sampling has *not* been evaluated at LKAB before and directed experiments are therefore critically needed, especially in the light that total sampling error effects often dominate the total uncertainty budgets by factors of 5-25, or more. This paper is a summary of the first experiments to advance in this direction.

1.2. Current practice for water sampling at LKAB

The current practice for water sampling at LKAB is based on sampling methods advised by the Swedish environmental authorities. The scope of the control program (e.g. frequency of sampling) used for self-monitoring varies between mining sites as this is governed by the individual permits issued for each site. The environmental permit for LKAB operations in Svappavaara stipulates that two surface water samples per week from the sampling target SVA, shall be collected and analysed. Even though specific days for sampling are not regulated in the permit, the sampling is normally performed every Tuesday and Thursday, due to practical reasons such as sample transportation and laboratory hours. For LKAB operations in Kiruna, the environmental permit stipulates extraction and analysis of one surface water sample per month from the sampling target KVA. The sampling protocol specify the week for the sampling to take place, but not a specific day. Most of the self-monitoring surface water samples at LKAB are planned annually on a weekly or monthly basis as with KVA. This gives the sampling technicians some freedom to plan their working hours according to other tasks as well as favourable weather conditions.

Both sampling targets, KVA and SVA, are located in smaller streams, approximately 10-15m wide. The KVA sampling target is at the beginning of an artificial dike which serve as the outlet from the clarification pond, leading the water to the lake Mettä Rakkurijärvi the recipient water system, Figure 1a. Approximately 98% of the water in KVA is discharge from the clarification pond, while only 2% is natural flow from precipitation and ground water. The SVA sampling target is in the natural stream Liukattijoki, which periodically receives discharge water from the water equalising storage (Metträsket) which in turn receives water from the tailings dam via the clarification pond (the water is pumped in pipelines from the clarification pond to Metträsket), Figure 1b. The discharge from Metträsket to Liukattijoki varies between 0-50% in relation to the natural flow in the stream. The discharge can be momentarily high, but no continuous outflow is present, meaning that the concentrations at SVA is mainly related to the level of discharge in relation to the natural flow in Liukattijoki. The highest ratio of discharge water occurs when the natural flow is low and a large release from Metträsket is initiated. This means that the absolute analyte concentrations in the clarification pond does not affect the concentrations at SVA to the same degree as it does at KVA. In Kiruna, the discharge from the clarification pond to the artificial dike is more uniform over time, as well as contributing to the major part, ≈98%, of the stream water at the KVA sampling target.

The objective of the measurement systems at both SVA and KVA is to monitor the analyte concentrations released to the recipient streams. The goal is therefore not to identify peak concentrations, but rather to monitor the mean concentration (and total amount) being released from the process to the recipient water system. The environmental permits stipulate the maximum allowable concentrations reached in the recipient streams, rather than the concentrations being released, meaning that monitoring of natural flow in the recipient is an important complement to the chemical analysis of the sampled water.

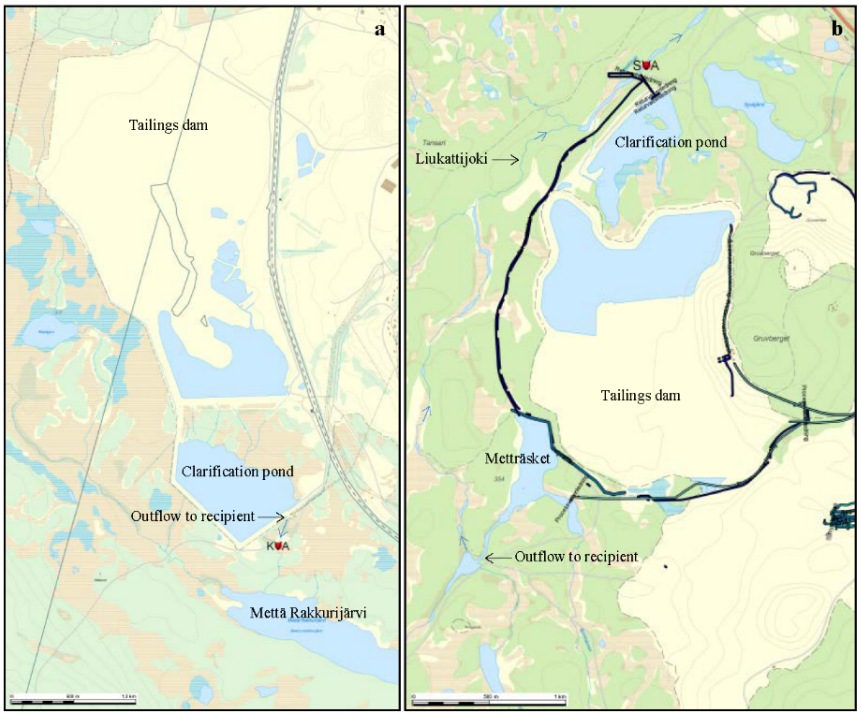

Fig. 1 Water systems in a) Kiruna and b) Svappavaara, with tailings dams, clarifications ponds, Mettträsket, Mettä Rakkurijärvi, outflow location and sampling targets KVA and SVA annotated. The black lines in 1b corresponds to the pipelines pumping water from e.g. the clarification pond to Mettträsket.

1.3. General and local variability in recipient stream water

Diurnal, seasonal and event-driven variability of water quality is present in any stream or other water catchment. These variations are for example dependent on air-temperature, solar radiation, photosynthesis, respiration, stream flow variation, circulation and photochemical processes (Henjum et al. 2010). Bio-geochemical processes can be affected by the solar photo-cycle and the level of diurnal scale variation can be as large as annual or seasonal time scales. The 24-hour variations in metal concentrations has also been observed to be larger than long term, e.g. seasonal variations (Nimick et al. 2005). Metal concentrations in stream water can vary in relation to the downstream distance from the primary source and has been shown to behave differently depending on the contaminant being studied. As the diurnal and groundwater variabilities also is significant for the metal contaminants, these variabilities need to be addressed in relation to each other (Gammons et al. 2007).

The size of the clarification pond in Kiruna is 2.3 Mm^3, and the process water inflow to the tailings dams and clarification ponds is close to uniform over time in both Kiruna and Svappavaara. The changes in inflow of water is therefore mainly affected by snow-melting and precipitation. The seasonal variability of natural flow in the recipient water streams is large and similarly dependent on snow-melting and precipitation. Flow peaks are generally registered during the spring flood and during heavy rains in the summer, while the snow-covered period in winter leads to significantly lower flows (Westerberg and Huseby Karlsen, 2017). In Kiruna, increased inflow to the clarification pond leads to a direct increase of outflow to the recipient water system, as the overflow is controlled by a fixed threshold. The clarification pond in Svappavaara is significantly smaller than in Kiruna (0.3 m^3) and is therefore followed by an equalising water storage pond, Mettträsket, for additional storage capacities. The outflow from the clarification pond runs via Mettträsket and is controlled relative to the water level in Mettträsket. This system leads to an uneven outflow from the internal water system to the recipient stream, Liukattijoki, as hatches are opened to release larger amounts of water when the level in Mettträsket

reaches a predetermined set point. This results in momentarily large water emissions, rather than a regular outflow over longer time periods. Therefore, the concentration variations in Liukattijoki is largely dependent on the outflow flux from the water storage in relation to the natural flow in the stream, which in turn exhibit large seasonal variations, from ≈0.2 to ≈8 m3 per second (Westerberg and Huseby Karlsen, 2017). An important limitation for the control of the outflow in both Kiruna and Svappavaara is the restricted storage capacity in the clarification ponds and Mettträsket. A regulated overflow, related to the natural flow in the recipient streams, would be preferred to even out the analytes concentrations in the recipient water system. However, this is currently not possible as the overflow must be regulated in relation to the storage capacity in the ponds.

The circulation in the LKAB clarification ponds is continuously high due to the large inflow of process water via the tailings dam. This is coupled with water being pumped back to the processing plants from the outflow of the clarification pond. The high circulation in the water system means that the time for sedimentation in the clarification pond is limited. As the circulation is mainly driven by the exchange of process water, the effects of ice coverage and wind is negligible in comparison.

The analyte concentrations in the discharged water from the clarification ponds in Kiruna and Svappavaara are not directly related to the viability of water inflow and level of dilution. For some analytes, e.g. nitrogen, dilution is visible during high precipitation. However, for sulphate, no direct dilution is visible as a chemical equilibrium is present in the clarification pond. This leads to sulphate being dissolved from the sediments in to the water if increased precipitation lowers the sulphate concentration in the pond. The dissolved sulphate will raise the concentration in the pond so that the equilibrium is retained, meaning that the sulphate concentration is not directly affected by the dilution during high precipitation. However, the dilution factor for sulphate is dependent on the water temperature, resulting in lower sulphate concentrations in the winter (with water temperatures close to 0°C) than in summer (Ylipää and Björnfot, 2018). Another reason for seasonal variation is biochemical process in the clarification pond, e.g. bacterial denitrification, that increases during the warm summer season with increased sunlight. Denitrification leads to a lowered concentration of nitrate during the warm seasons compared to the winter season. Internal calculations indicate that there is approximately a 20% decrease in nitrate emissions to the recipient water in Kiruna due to denitrification during the summer months (Ylipää and Björnfot, 2018).

1.4. Methods for evaluating sampling variability

Several methods, using both empirical and modelling approaches for evaluation of sampling and analytical uncertainties in environmental monitoring have been published recently (Guigues et al. 2016; Lyn et al. 2007; Ramsey et al. 2011). One recommended approach is the *duplicate method*, as it is both simple and cost-effective. The approach of the duplicate method is that the field sampler collects duplicate primary samples from 10% (but no less than eight samples) of the sampling targets (Grøn et al. 2007). To enable evaluation of the complete uncertainty, including primary sampling, possible sub-sampling and sample preparation, it is vital to duplicate the primary sample extraction, rather than only duplicating the sample preparation or sample analysis.

According to Ramsey et al. (2011) one important limitation of the *duplicate method* is that it excludes the uncertainty arising from *systematic sampling bias effects*. A method for inclusion of any so-called *systemic effects in sampling*, is the use of a sampling proficiency test (SPT). This method involves at least eight experienced samplers sampling from more than five typical sampling targets (Ramsey et al. 2011). An SPT using several samplers is expensive and often impossible to carry out due to proprietary reasons. It is therefore seldom practicable for validation of a sampling protocol in a single organization (Ramsey and Thompson 2007). The SPT approach has also been criticised by Esbensen and Wagner (2014), based on conceptual and theoretical problems with this approach; the main critique is the unrealistic concept of a "constant sampling bias" (Esbensen and Wagner 2014). Within TOS it has been shown that there does not exist a *constant sampling bias* or *a systematic sampling effect* (breaking with the *oft-assumed* analogy to the analytical bias). In fact, every time a sampling bias is estimated it will be different, i.e. the sampling bias is *inconstant* and *variable*, because of the irregular lot heterogeneity. A sampling bias (always random) must be reduced, preferentially eliminated, by specific application of all necessary elements in TOS that are aimed at achieving correct sampling. On this basis i.e. when a non-biased sampling process has been achieved, the use of duplicate and/or replication experiments will then be sufficient and effective for estimation of the complete sampling variability as manifested by $MU_{sampling+analysis}$ (Esbensen et al. 2007; Esbensen and Wagner 2016; Pitard 1993). As the aim of this paper is to

apply the concepts of TOS to environmental recipient water sampling, SPT is not an applicable approach, but correct duplicate and replication experiments are sufficient for estimation of all sampling error effects.

An important aspect when assessing the variability in sampling-and-analysis is setting an acceptance level below which one can claim a "fit-for-purpose" status. I.e. what is the relative measurement uncertainty, including variability stemming from both sampling and analysis, acceptable for receiving meaningful data that can be used for evaluation and decision making. Even though such thresholds need to be set in relation to the specific lot material and sampling procedure used, Ramsey et al. (1992) suggests that the total measurement variability (including sampling) should not contribute to more than 20% of the total variability of the analyte over time. In the sampling community, a general consensus indicates that a threshold of 20% for the relative sampling variability (i.e. the sampling variability in relation to the average analyte concentration) is also recommended here (Esbensen and Wagner 2016). However, TOS also strongly advocates individual assessment and determination of sampling variability thresholds, for each specific sampling procedure, due to the fact that sampling targets in general display significantly different heterogeneities (DS3077 2013).

1.5. Study objectives

The objectives of this study are to perform an initial evaluation of the sampling variability associated with the current LKAB environmental measurement system for emissions to surface water. With the use of the duplicate method and replication experiments, the sampling system for recipient water at the sampling targets KVA and SVA, will be evaluated and possible improvements will be suggested. For all reported experiments, the replication/duplication has been executed at the primary sample extraction so as to include all variability effects stemming from the complete sampling to analysis pathway. A second objective is to evaluate the experimental methods for estimation of sampling variability in recipient water sampling for environmental monitoring.

As the objective is to conduct initial evaluations of the sampling variability, together with evaluation of the specific experimental methods, the time frames for the experimental campaigns are limited. One consequence of this is that the complete seasonal variability in analyte concentrations and flow will not be covered. However, the study will provide recommendations for how to implement a comprehensive and continuous control of the sampling variability of surface water sampling at LKAB. Note that the study does not intend to evaluate the seasonal or yearly variability of analyte concentrations in the stream water, but is limited to studying the specific sampling variabilities of the current measurement systems in use.

2. Sampling and analytical methods

All sampling experiments were conducted on the sampling targets regularly used in the current self-monitoring programs at LKAB. The sampling target KVA in Kiruna and SVA in Svappavaara were selected as these are important for the environmental permits and have frequent historical data for all important analytical parameters. For the KVA sampling target, one duplicate, two replication and one variographic sampling experiment were conducted. For the SVA sampling target, only one duplicate and one replicate sampling experiment were conducted in this study.

The current physical extraction of water samples method used by LKAB was used during all experiments in this study. Within the TOS understanding, this approach is a *manual grab sampling* (usually rejected as it can never be representative) but it is the official method described and taught by water sampling consultants that follow the Swedish government regulations for environmental self-monitoring. The sample extraction is to be performed in the centre of the targeted stream, approximately 50 cm from the surface (or at mid depth if the stream is shallower than one metre). To be able to reach the centre of the stream, a sample container can be attached to an extension pole, Figure 2. To allow for a sufficient amount of water to be collected, three increments are collected from the same location and combined to one primary sample. The current grab sampling introduces the sampling errors IDE and IEE as the sample extraction does not allow equal probability for the complete lot (flux cross section) to end up in the sample. A TOS correct sample extraction requires that the complete cross section of the lot should be covered by the sampling equipment and that the speed with which it is crossing from one side of the stream to the other is uniform. Hence, if the volume or speed of water is less in the shallower water by the sides of the stream, the amount of water collected is less than in the centre of the stream where the flow is faster. Currently, it is impossible to achieve this proportional, TOS-correct sample extraction using the recommended sampling container.

Fig. 2 Extendable pole with standard plastic sample container used to collect water samples according to the official protocols in the present study

In an attempt to handle some of the ISE associated with the traditional grab sampling of stream water, Ramsey (2014) presented the Aloha Sampler™. The design of this sampling equipment, Figure 3, enable sample collection from the complete cross section of a reachable water stream as is the case at e.g. sampling target KVA. The small holes in the lid of the sampler allow for water to enter and an equivalent volume of air to exit in a controlled, slow fashion (the Aloha Sampler™ comes with optional hole diameters), enabling sampling staff to move the sampler and collect water across the stream in a continuous motion (Ramsey 2014). The Aloha Sampler™ was used in one replication experiment at the KVA sampling target for comparison with the current sample extraction method used by LKAB. The continuous sampling motion applied in this study, using the Aloha Sampler™, is visualised in Figure 4.

Fig. 3 The Aloha Sampler™ designed for composite sampling, for example along a complete cross section of the streaming water

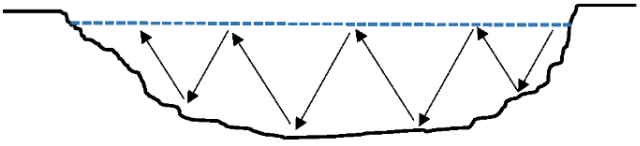

Fig. 4 Aloha Sampler™ used in a continuous motion to collect water from a complete cross section of a stream (Ramsey C 2014)

All water samples were analysed at the internal LKAB environmental laboratory in Kiruna or at ALS in Luleå. All analytical methods are accredited by official national accreditation agencies as to ensure valid analyses for the environmental permits. A list of analytical methods and location for analysis is presented in Table 1. Note that routine samples are analysed for several additional parameters. The ones presented in this study are selected as they are coupled with possible ecological effects in the recipient water and therefore important for the LKAB environmental permits.

Table 1 Location for analysis of the evaluated analytical parameters

	Analytical method	**LKAB laboratory Kiruna**	**External laboratory**
Alkalinity	ISO 9963-2 Water quality - Determination of alkalinity - Part 2: Determination of carbonate alkalinity	X	
Conductivity	SS-EN 27 888 Water quality – Determination of electrical conductivity (ISO 7888:1985)	X	
Total Phosphorus	SS-EN ISO 6878:2005 Water quality – Determination of phosphorus – Ammonium molybdate spectrometric method (ISO 6878:2004)	X	
Nitrate Sulphate	SS-EN ISO 10304-1:2009 Water quality – Determination of dissolved anions by liquid chromatography of ions – Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulphate (ISO 10304-1:2007)		X
Copper Vanadium Zinc Uranium	SS EN ISO 17294-1, 2 (mod) / EPA-method 200.8 (mod) Water quality – Application of inductively coupled plasma mass spectrometry (ICP-MS)		X

2.1. Duplicate sampling experiment

The *duplicate method* is the most straight forward method for assessing the variability stemming from sampling, sub-sampling and analysis in relation to the overall variability in the data (Ramsey 1998). According to Lyn et al. (2007) the duplicates should be collected in accordance to the current sampling protocol, reflecting all ambiguity that can be expected. This means that duplicates are separated in time and/or space by the same ambiguity that is presented by the sampling protocol. The ambiguity of a sampling protocol leads to diverse interpretation or application of the protocol in different situations, e.g. variations in the sample extraction location or variations in the time for sample extraction. The primary sample duplicates should ideally also be analysed in duplicates to enable the use of Analysis of Variance (ANOVA) for separation and quantification of the variability stemming from sampling and analysis respectively (Lyn et al. 2007). It is important to clearly describe the exact step where duplication is applied to allow clear indication of which variabilities are included in each variance estimation. To accommodate for possible outlying values, the robust ANOVA (RANOVA) is the recommended statistical method to use. The classic ANOVA is sensitive to outlying values due to sampling, geochemical or technical issues (Ramsey et al. 1992). However, the robust method is claimed to provide valid estimates of the variability components 'under average background levels'. For example, single outliers do not affect the results of a RANOVA unduly, like it would classic ANOVA (Ramsey et al. 1992). There are serious misgivings in the TOS community regarding this position regarding outliers.

Two duplicate sampling experiments were carried out at the KVA and SVA sampling targets respectively. The KVA duplicate sampling experiment was carried out in accordance to the method described by Lyn et al. (2007), which stipulate that the distance between the duplicated samples in time or space should be separated according to the ambiguity in the sampling protocol. Especially for sampling targets with large temporal variability, e.g. stream water as is the case for both KVA and SVA, the periodic separation between duplicates should correspond to the uncertainty in time that is described by the sampling protocol (Ramsey 1998). In this case, the KVA sampling protocol specify a fixed week for sampling once every month, while the day for sampling is open within that week. This means that the ambiguity in the KVA sampling protocol can lead to a temporal spacing between routine samples of up to six days (within one week). To reflect the possible variability *within* the

sampling protocol, this sampling experiment extracted duplicates with *random time spacing* between one and six days. Each duplicate sample was analysed in duplicate for the important parameters, including Alkalinity, Conductivity, Phosphorus, Nitrate, Sulphate, Copper, Vanadium, Zinc and Uranium. The experiment was conducted during eight consecutive weeks resulting in a total of eight duplicate samples and 32 individual assay results per parameter.

As SVA is sampled twice a week, routinely on specific days, the sampling protocol is more precise compared to KVA. The SVA experiment was carried out by extracting duplicate samples under *repeatability conditions*, according to the definition in Grøn et al. (2007, p.29). Repeatability conditions are here defined so as the samples are collected at the *same* time, by the *same* person, at the *same* sampling target, using the same sampling equipment. This will result in a minimum variability introduced by the primary sample extraction, excluding effects related to long term temporal changes in the quality of sampled water or changes in sampling staff or sample equipment.

For the SVA duplicate samples, no duplicate analysis was conducted. However, as the samples go through identical sample preparation and laboratory analysis at the same laboratories as the KVA samples, the analytical uncertainty can be assumed equal as these samples. The duplicate samples were collected once a week (at every other routine sampling extraction) for 15 weeks, resulting in 15 paired samples.

2.2. Replication sampling experiment

Replication experiments (RE) are used to calculate the relative sampling variability (RSV), a sampling quality index describing the variability for a specific sampling and analysis procedure (DS3077 2013). The RE is particularly useful when sampling stationary lots, in comparison to variographic characterization, which is more powerful when assessing measurement systems performance for dynamic lots in process control (Esbensen and Wagner 2016). The RE is a practical and informative method for both initial and regular quality assessment of recipient water sampling.

In this study, the sampling of SVA targets were replicated using the current sample extraction method and for KVA, both the current sample extraction and the Aloha Sampler™ were used in two separate RE. During the experiment, ten replicate primary samples were collected in direct repetition and brought to the laboratory for analysis. The objective for not accounting for the temporal ambiguity in the sampling protocols was to enable direct comparison of the results from the two separate sampling targets (KVA/SVA) and the two alternative sampling methods (current/Aloha Sampler™). The complete primary sampling was replicated, meaning that for each of the ten replicates, three primary increments were collected and combined to one primary composite sample. For one of the replicate samples, the analysis was also replicated ten times for the parameters: Alkalinity, Conductivity and Total Phosphorus. This allows for estimation of both the RSV as well as the isolated analytical variability under repeatability conditions.

2.3. Expression of results

The relative standard deviation for sampling (s_s) and analysis (s_a) respectively, as well as the total variability for the measurement system (s_m) is calculated for the evaluated sampling targets, equation 1. It is common to present uncertainties as a relative *expanded uncertainty* (U), with a *coverage factor* of k = 2, equation 2 (Ramsey 1998). Under the assumption of normally distributed data this results in an uncertainty that describes a 95% confidence interval for the average result ($\bar{x}$). In this study, results will be presented as relative expanded uncertainties, denoted U, with a uniform coverage factor of 2. The relative uncertainty is especially useful for comparison between different analytes and different experimental methods.

$$s_m = \sqrt{{s_s}^2 + {s_a}^2} \tag{1}$$

$$U\,\% = 100\frac{2s}{\bar{x}}\,\% \tag{2}$$

3. Results

3.1. Duplicate sampling experiment

The results from the two separate duplication experiments in Kiruna and Svappavaara showed different sampling variability. The variabilities observed for KVA were significantly higher than the variability at SVA, for all analysed parameters, Table 2. The absolute concentration for all analytes are also higher for KVA than for SVA. The higher concentrations for KVA is because the water at KVA is mainly water from the clarification pond, while SVA is a pre-existing stream with mostly natural water and only temporary overflow from the clarification pond. The water in the clarification pond preceding KVA also has higher concentrations of most elements as the Kiruna ore is richer in salts and the processing water is more recirculated in the Kiruna processing plants. Note that the expanded relative uncertainty from sampling, for sampling target SVA, is calculated from the total measurement uncertainty for SVA and the uncertainty from analysis for KVA sampling target (as no duplicate analysis was conducted for SVA). This will have no practical implications on the evaluation as the sampling preparation and analytical process is identical for both sampling targets (i.e. the primary samples are delivered to the same laboratory and are prepared and analysed by the same personnel, using identical methods and equipment for all incoming water samples).

The results from the duplicate experiment show that the measurement variability (including sampling), in relation to the total variability, is less than 20% for SVA. This indicates that the measurement variability is within recommended limits for this sampling target. For KVA, the total measurement variability (including sampling), in relation to the total variability, ranges 15-80% for the analysed parameters. Alkalinity, Conductivity and Phosphorus are within the requirement of less than 20%, while the variability for the remaining parameters exceeds this threshold. The main reason for the differences is that the duplicates at SVA were collected under *repeatability conditions* (as a reflection of the smaller ambiguity in the sampling protocol for SVA), while the temporal separation of duplicates at KVA was up to six days (as a reflection of the larger ambiguity in the sampling protocol for KVA). The temporal ambiguity in the sampling protocol for KVA, translated to the duplicate experiment, means that the variations in absolute analyte concentrations in the stream water, over six days, are included in the measurement system variability assessment. This is a direct consequence of the KVA sampling protocol that allows large ambiguity in the time for sample extraction. Comparatively, the sampling protocol for SVA has a more strict time spacing, where two samples are to be collected every week and these are routinely collected the same weekdays, i.e. the temporal ambiguity is significantly smaller for the SVA sampling protocol and the measurement variability in relation to the total variability will therefore also be significantly lower.

Table 2 Variability results for duplicate sampling experiments for SVA and KVA sampling targets

		$U_{a\text{-}lab}$	$U_{a\text{-}dupl}$	$U_{s\text{-}dup}$		$U_{m\text{-}dup}$		rel meas var		Mean	
Chemical parameter	**Unit**	**Expanded relative uncertainty from Analysis (incl. bias)** (95%) (From laboratory QA/QC program)	**Expanded realtive uncertainty from analysis** (95%) **RANOVA of duplicates**	**Expanded realtive uncertainty from Sampling** (95%) **RANOVA of duplicates**		**Expanded realtive uncertainty from measurement** (95%) (sampling + analysis) **RANOVA of duplicates**		**% of total variance for measurement** (sampling + analysis) **RANOVA of duplicates**		**Mean value/concentration during experiment**	
			KVA	SVA	KVA	SVA	KVA	SVA	KVA	SVA	KVA
Alkalinity	mmol/l	10%	0.0%	-	3.2%	-	3.2%	-	20.9%	-	48.7
Conductivity	mS/m	5%	0.5%	0.2%	0.6%	0.6%	0.8%	0%	13.7%	23.3	284.1
P_{tot}	mg/l	25%	7.6%	-	18.1%	-	19.6%	-	14.9%	-	0.028
Nitrate	mg/l	15%	3.1%	< 0	8.7%	1.5%	9.2%	0.01%	36.1%	0.70	19.41
Sulphate	mg/l	15%	6.8%	< 0	28.0%	2.0%	28.8%	0.02%	79.6%	60.3	1369
Copper	µg/l	≈ 20%	8.2%	15.6%	34.1%	17.6%	35.1%	10.4%	67.1%	0.60	1.75
Vanadium	µg/l	≈ 20%	4.0%	12.3%	27.2%	13.0%	27.5%	3.0%	49.4%	0.12	2.49
Zinc	µg/l	≈ 20%	32.3%	32.0%	58.4%	45.5%	66.7%	8.6%	30.0%	2.48	3.32
Uranium	µg/l	≈ 20%	6.0%	< 0	17.8%	5.0%	18.8%	0.18%	31.8%	0.72	13.1

3.2. Replication sampling experiment

The results from the three replication experiments (SVA, KVA, KVA_{AS}) indicate that the relative measurement variabilities (including sampling and analysis) for all analysed parameters are below the 20% threshold, Table 3. For the three common parameters (conductivity, Nitrate and Sulphate) both sampling targets show similar results for the traditional sampling method used by LKAB, despite the large difference in absolute concentration. The remaining six parameters were only analysed for one of the two sampling targets as a direct reflection of the two separate regulatory sampling protocols requiring different parameters.

However, the duplicate experiment at KVA show that the large temporal variability in the stream water increase the measurement variability significantly when duplicates are spaced further apart. Furthermore, the results from the Aloha Sampler™ replication experiment (KVA_{AS}), Table 3, showed slightly higher total measurement variability compared to the traditional sample extraction method. This could partly be due to that the continuous motion of the Aloha Sampler™, collecting an entire cross section of the stream, which will allow for a wider coverage of the overall heterogeneity in the streaming water. A possible influence for the Aloha Sampler™ procedure is its up-and-down motion, which results in a differential influence due to the hydrostatic pressure in the stream; more water will be forced into the sampling container as depth increases. If the evaluated analyte is unevenly distributed with depth, this could therefore result in higher variability for the Aloha Sampler™ approach relative to stationary grab sampling. The increment collection is also slower than for the traditional grab sampling from the centre of the stream. This means that the time frame for collecting all 10 replicate samples increase from approximately 15 minutes with the traditional method, compared to one hour for the Aloha Sampler™. This fourfold increase in duration will allow larger temporal variability in stream flux to influence the sampling, which perhaps could be another reason for the slight increase in relative measurement uncertainty when using the Aloha Sampler™ for primary sample extraction.

Table 3 Variability results for replication sampling experiments for SVA and KVA sampling targets. KVA_{AS} represent the results from the replication experiment using the Aloha Sampler™ at KVA sampling target

		$U_{a\text{-}lab}$	$U_{a\text{-}dupl}$	$U_{s\text{-}dup}$		$U_{m\text{-}dup}$			% meas var			Mean	
Chemical parameter	Unit	Expanded relative uncertainty from Analysis (incl. bias) (95%) (From laboratory QA/QC program)	Expanded realtive uncertainty from analysis (95%) COV of laboratory replication	Expanded realtive uncertainty from Sampling (95%) RANOVA of duplicate exp.		Expanded realtive uncertainty from measurement (95%) (sampling + analysis) COV of replication exp.			Relative measurement variability (sampling + analysis) COV of replication exp.			Mean value/concentration during experiment	
			KVA	SVA	KVA	SVA	KVA	KVA_{AS}	SVA	KVA	KVA_{AS}	SVA	KVA
Alkalinity	mmol/l	10%	1.2%	< 0	2.3%	-	2.6%	0.0%	-	1.3%	0.0%	-	0.4
Conductivity	mS/m	5%	0.4%	0.9%	0.3%	1.0%	0.5%	0.7%	0.5%	0.2%	0.3%	9.9	292
P_{tot}	mg/l	25%	24.7%	< 0	< 0	-	9.8%	15.6%	-	4.9%	7.8%	-	0.027
Nitrate	mg/l	15%	-	-	-	3.1%	2.9%	4.0%	1.5%	1.4%	2.0%	0.13	23.04
Sulphate	mg/l	15%	-	-	-	7.3%	4.7%	7.3%	3.7%	2.3%	3.6%	17.2	1217
Copper	µg/l	≈ 20%	-	-	-	29.5%	-	-	14.7%	-	-	0.39	-
Vanadium	µg/l	≈ 20%	-	-	-	23.1%	-	-	11.5%	-	-	0.08	-
Zinc	µg/l	≈ 20%	-	-	-	28.5%	-	-	14.2%	-	-	4.26	-
Uranium	µg/l	≈ 20%	-	-	-	9.2%	-	-	4.6%	-	-	0.44	-

4. Discussion

The results from this study are explicitly showing how important the experimental design is for proper evaluation of the different sampling system variabilities. The design and adherence to guidelines and norms will directly influence the numerical evaluation of the results and therefore need to be fully transparent and well defined. The implication of the results from the two separate duplicate sampling experiments, indicate that the current water sampling and analytical process is showing acceptable variability *under repeatability conditions*, or as described by a strictly defined sampling protocol. However, the temporal spacing between primary samples at KVA is leading to high measurement system variability, due to the large temporal variabilities in the stream water. This study shows that replication experiments together with duplicate sampling under repeatability conditions, will lead to lower sampling and measurement variability estimations, than duplicate experiments, taking the ambiguity in the sampling protocol in to account. The main reason for this is the large temporal changes at the sampling locations, stemming from e.g. diurnal variabilities in the water.

The replication experiment using the Aloha Sampler™ showed higher sampling variability than the current grab sampling method for all parameters except alkalinity. This is likely due to that the Aloha Sampler™ covers a larger part of the cross-stream heterogeneity and collects increments during longer periods of time. The reason that this does not increase the measurement variability for alkalinity is likely due to the lower overall variability for this parameter, compared to all other analytes. The spatial variability for alkalinity might be larger than the temporal variability, leading to larger variabilities for the grab sampling approach as applied to one more-or-less randomly selected location in the stream, than for the Aloha Sampler™ (collecting increments from the complete width of the stream). One advantage of using the Aloha Sampler™ for routine sampling would a wider coverage of the stream width, as well as covering longer time intervals and thereby temporal concentration changes. With the existing diurnal and seasonal changes in analyte concentration and outflow from the clarification pond, this small extension in time coverage would likely still lead to large sampling variabilities when the temporal ambiguity in the current sampling protocol is taken into account.

It is not only the practical sample extraction method that needs to be evaluated to determine the complete measurement variability. The environmental sampling protocols, dictating *when* and *how often* samples shall be extracted, are just as important. This study gives a clear indication that the temporal variabilities in natural stream flow, outflow from the industrial operation and analyte concentrations, need to be taken into account when designing improved sampling protocols, both in regard to sampling interval and sample extraction method. It is essential to ensure that the variability in the 'true' concentrations are intercepted and represented correctly. For sampling targets with large variabilities in concentration and stream flow, or variabilities in overflow from e.g. a clarification pond, the sampling protocol must be adapted to these empirical conditions. The sampling and measurement variability for especially KVA could be improved by a more concise sampling protocol that *counteracts* the temporal variabilities. One recommended solution would be to install an automated sampler, collecting increments from the stream, either at regular time intervals or based on *volume-proportional composite sampling*. An important learning experience from TOS, also revealed in this study, is that standards and norm-giving manuals not necessarily guarantee that recommended sampling approaches are correct or representative. The evaluation of total measurement variability through well designed experiments are therefore always necessary.

To allow for continuous control of the current sampling systems for recipient water sampling, systematic duplication or replication experiments at regular time intervals is recommended. However, the ambiguity in existing sampling protocols, as described by e.g. Esbensen and Wagner (2016), Lyn et al. (2007) and Ramsey (1998), must be taken in to account to allow valid estimations of the complete sampling and measurement variability. In comparison, to follow the duplicate method as described by Grøn et al. (2007) strictly (extracting duplicates specifically under repeatable conditions) will unavoidably *underestimate* the complete sampling variability derived by most sampling protocols.

5. Conclusions

This study shows that the specific approach for estimating sampling variability will affect the results significantly. The scope of each specific investigation (experimental design, time duration, sampling frequency) must be carefully defined so that the most appropriate experimental method, that will best match the pre-set objectives, can be selected. This study has concluded that replication experiments together with duplicate sampling under repeatability conditions, will lead to lower sampling and measurement variability estimations, than duplicate experiments taking the ambiguity in the sampling protocol in to account. Hence, to allow correct evaluation of all variabilities affecting the measurement system, all ambiguities in the sampling protocol need to be *included* in the experimental design. The time-varying nature of stream water makes sampling a complex undertaking, for which the existing official protocols is in need of revision.

The present results indicate that the relative sampling-and-measurement variability is acceptable under repeatability conditions, both using the current sample extraction method and using the Aloha Sampler™. However, the duplicate experiment at KVA, where the larger ambiguity in the sampling protocol was taken in to account, indicated that the measurement variability for several analytes were above the 20 % threshold. This shows that even if the sample extraction can produce repeatable results over short times periods, ill-reflected ambiguity in the current sampling protocol for KVA is leading to adversely large measurement variabilities for some analytes. The results of this study do not enable definite conclusions regarding if the Aloha Sampler™ is more reliable than the traditional grab sampling. The Aloha Sampler™ replication experiments shows larger sampling variability (for most analytes) compared to the current sample extraction method, but this estimate is likely more realistic.

A recommended solution for counteracting the large temporal variabilities is to install automated samplers, regularly collecting increments from the stream and combining them to composite samples. With a composite sampling method and regular analysis, the coverage of temporal changes in stream flow and analyte concentration would be significantly improved, compared to any manual sampling approach. Both duplicate and/or replication experiment (where samples are spaced in relation to the ambiguity in the sampling protocol) is concluded to be practical and rewarding methods for initial, as well as regular, quality assessment of recipient water sampling. To allow for a complete evaluation of the sampling and analysis variability, representative for all seasonal variations in flow and analyte concentrations, the experiments should be applied continuously and regularly over a whole year.

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Paper V

Evaluation of sampling systems in iron ore concentrating and pelletizing processes – Quantification of Total Sampling Error (TSE) vs. process variation

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Evaluation of sampling systems in iron ore concentrating and pelletizing processes – Quantification of Total Sampling Error (TSE) vs. process variation

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ABSTRACT

Process sampling is involved in grade control in all parts of the production value chain in mineral processing. Reliable sampling and assaying is essential to ensure final product quality, but the need for representative sampling is not always taken into account. By continuous control of the variability of sampling systems, a comprehensive understanding of the relationship between the sampling and process variability can lower the risk for overcorrections of process parameters due to sampling variability rather than process changes. Variographic characterization of process sampling makes it possible to assess all combined sampling and analytical errors from only 40–60 routine analytical values. The objective of this study is to evaluate total sampling variability in relation to process variability in the concentrating and pelletizing process sampling at LKAB. The results from the variographic analyses will form a basis for suggestions of possible improvements. The results show that variographic analysis is a powerful tool to evaluate both process variations and the variability of the sampling systems employed. The extensive access to time series data allow variographic characterization (quality control) of all critical measurement systems and locations. At the same time, periodicity and small changes in process variation can be detected and counteracted early, minimizing the risk for producing products out of specification.

1. Introduction

Process sampling is used for grade control in all parts of the production value chain in the minerals industry. Samples are extracted from slurry pipes, blender tanks, conveyor belts, stock piles and more, not always taking into account the need for representative sampling however. The process of sampling and assaying in all stages of sorting, concentrating and pelletizing is essential to ensure correct quality of the final product. One important aspect to reach correct quality within customer specifications is to be able to control the production process without overcorrecting for variability that derives from the sampling system rather than the process. The risk for this rises as the specification limits closes in on the variability of the sampling system itself. By continuous control of sampling systems, through variographic analysis, a comprehensive understanding of the relationship between the sampling - and process variability can lower the risk for overcorrections leading to unnecessary process changes (Minnitt and Pitard, 2008). Without meaningful data there is little point in trying to draw correct and reliable conclusions (Esbensen and Paasch-Mortensen, 2010). Cost, rather than the request for representative samples, is often the driving force for decision making by company management (Holmes, 2004, 2010). To be able to make these decisions correctly, there is however a need to fully understand the performance of the current process sampling systems in relation to the process variability.

There is no possibility to assess if a particular sample is representative by only looking at the sample itself (DS 3077, 2013), only the sampling process can be graded as representative or not. For one dimensional lots, i.e. process streams (Pitard, 1993), Theory of Sampling (TOS) offers variographics to fully characterize both process variability as well as all combined sampling and analytical errors generated by the complete sampling system. The variographic characterization of the process sampling makes it possible to assess the Total Sampling Error (TSE) from only 40–60 routine process analytical values (Esbensen et al., 2007).

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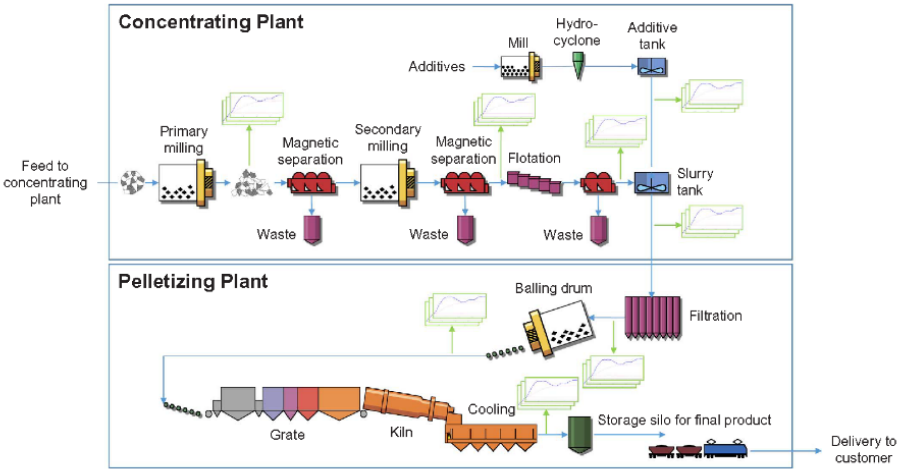

Fig. 1. Overview of the LKAB concentrating and pelletizing process. Sampling locations evaluated through variographic characterization are indicated.

1.1. Objectives

Luossavaara-Kiirunavaara limited company (LKAB) is a state owned mining company in the north of Sweden. The core business is producing high quality iron ore pellets for blast furnace and direct reduction steel making. Quality control in all parts of the concentrating and pelletizing process is important and continuous sampling and analysis is performed at all important process steps. Important quality characteristics for process control include iron and silica grade, moisture content and pellet size distribution.

The objective of this study is to evaluate TSE in the concentrating and pelletizing process sampling at LKAB with variographic characterization, see Fig. 1. By the use of existing process data, the performance of the sampling systems will be evaluated with regards to sampling variability in relation to process variability and control. The results from the variographic analyses will form a basis for suggestions of possible improvements of the evaluated sampling systems. The general theoretical methodology for variographic characterization, as well as the calculation of the relative semi variogram is fully described by e.g. Minnitt and Esbensen (2017), Esbensen and Paasch-Mortensen (2010), Minnitt and Pitard (2008), Pitard (1993), and will therefore not be described in detail here. The objective of the present study is to exemplify the practical application of variographics in a large-scale mineral processing industry; iron ore concentrating and pelletizing, illustrating the possibilities for improved process interpretation and control.

The main processes of LKAB is described by Fig. 1. Three stages of milling and magnetic separation is followed by floatation to remove phosphorus. A final magnetic separation stage is performed to reach desired iron grade. The magnetite slurry is mixed with specific additives dependent on final product and filtered to correct moisture content for successful balling in balling drums. After sieving, the correct size distribution of raw pellets is sintered in the grate-kiln oven. The final product is stored in silos and loaded to trains for delivery to the harbour and further sea transport to customer.

2. Theory

Variography is a powerful tool for the study of serial datasets. With the use of the relative semi-variogram all forms of heterogeneity fluctuations or variability present in the data can be quantified, short range (random discontinuous term), long range (trend term) and periodic (cyclic term) variabilities (Pitard, 1993). Variographics allow separation of variability stemming from the sampling system from the true process variability. The so called nugget effect of the variogram is defined by extrapolation to the y-axis intersect of the variogram. This value is an important parameter as it is a estimation of the total error variance of the current measurement system (Minkkinen, 2013; Gy 1999). The nugget effect is termed V(0) and is often calculated by extrapolation of the variogram to the intersect of the y-axis. In chronostatistics, i.e. the process variogram, the nugget encompasses the short range random variability from sampling, preparation and measurement (Pitard, 1993). These total measurement system error contributions can be subtracted from the observable process variability in order to gain insight into the true process variability.

A telling aspect of the variogram is the ratio between the nugget effect and the sill, expressing the fraction of the observable process variability made up by the measurement system error (Esbensen and Paasch-Mortensen, 2010). If the sampling system accounts for a major part of the total variability (above 20–25%), the possibilities for successful process control are reduced, as the actual process variations are increasingly covered by the variability (noise) of the sampling system (Minnitt and Esbensen, 2017; Minnitt and Pitard, 2008). The most important parameters of the semi-variogram are presented in Table 1.

The method of variographic characterization of sequential data, including calculation of relative semi-variograms has been described in numerous reference publications, see e.g. Minnitt and Esbensen (2017), Esbensen and Paasch-Mortensen (2010), Minnitt and Pitard (2008) and Pitard (1993) and need no detailed description here.

In this study, relative variograms have been calculated for existing process control data with the purpose of evaluating precision of the sampling systems in concentrating and pelletizing plants at LKAB and to assess the possibilities to control the process with the use of current sampling systems. Possible improvements or necessary further

Table 1
Important parameters characterizing the relative variogram, e.g. Petersen et al. (2005), Minnitt and Pitard (2008).

Notation	Definition	Description
V(0)	Extrapolated intersect of y-axis	Short range random variability stemming from the sampling system, including sub-sampling, sample preparation and measurement. The nugget effect summarises both correct (CSE) and incorrect sampling errors (ISE), as well as the Total Analytical Error (TAE)
V(1)	Value at V(1)	The total variability representing the time period between two samples, including process and sampling variability
V(Process)	V(1) – V(0)	Process variability for the time period between two samples, not including the sampling system variability. The relationship between V(process) and V(0) is a good indication of the possibility of controlling the process with current sampling interval
Sill	Highest level of the variogram	Describes the highest variability, or the global heterogeneity, in the data series
Range	Where the variogram reaches the sill	Describes the lag distance outside which no autocorrelation can be detected. For process variograms the range can be directly translated into a time interval

variographic experiments will be suggested, together with a general methodology for continuous verification of sampling systems. This paper does not discuss the specific iron ore concentrating and pelletizing processes in detail, nor which analytic parameters are important or how they are used, as these are proprietary matters for LKAB.

3. Results

The variographic characterization has been applied to all sampling systems in one of the concentrating and pelletizing plants at LKAB. The selected sampling points evaluated are presented in Table 2.

In this paper, a selection of four typical variographic characterizations will be presented. The variograms are typical for the specific sampling point. Note that for all evaluated sampling points, several variograms have been prepared to ascertain that the results are similar for consecutive time periods (not shown here).

3.1. Iron grade after primary milling

A variogram for iron grade after primary milling in the concentrating plant is presented in Fig. 2. The sampling method for this analysis is a small manual grab sample taken every 8 h from a large slurry flow. The variogram show a high nugget effect to sill ratio of approximately 80%. This means that out of all the variability present in the grade control charts, 80% of the variability stems from the sampling system while only 20% of the variability represents the actual process variation. This indicates that the possibilities for successful control of the process or yield calculations using this sampling system are small indeed.

3.1.1. Interpretation and improvements

Manual grab sampling from a large slurry flow generates several correct and incorrect sampling errors which is clearly visible in the variographic characterization. The result is that the analytical results have a large uncertainty with respect to the moving slurry stream. When used for reconciliation purposes in the concentrating plant, this leads to calculations strongly affected by the sampling variability and bias. At this sampling point, there are alternative possibilities to extract a sample through round pumping from a distribution box (significantly larger primary sample). Even though this would not completely TOS correct, the increased mass would lead to a decrease in TSE, compared to the current sampling approach. When implementation of this sampling method is in place, a renewed variographic experiment would be appropriate to determine suitable sampling interval and evaluate the improved sampling performance.

3.2. Magnetite slurry iron grade

Figs. 3 and 4 are variograms for the same sampling system for iron grade of magnetite slurry. The completely automated sampling system consist of a shark fin sampler collecting a part of the slurry flow cross section all of the time. The primary slurry sample is split and after drying a secondary mass reduction is performed. After the second division of dried material, the subsample is transported by pneumatic dispatch to an automated XRF laboratory for analysis of iron grade and other important chemical parameters. For this sampling system, two variograms representing different time periods have different characteristics, see below. Both have a similar nugget effect and nugget to sill ratio of approximately 3–5%. However, one of the variograms is a classic increasing variogram with a clear sill and a range of about 24–30 lags (12 h). The second variogram have a periodicity of about 20 lags (8–10 h). This indicates a cyclic behaviour of the process in regards to iron grade. Some time periods indicate a cyclic behaviour of the process while other time periods has no periodicity what so ever. These examples show that through the use of variograms, cyclic behaviours of the process are more easily detectable then by only using the process control data alone.

3.2.1. Interpretation and improvements

The low nugget to sill ratio of 3–5% shows that the sampling system has an acceptable precision for the purpose of iron grade analysis. The sampling system is not TOS compliant as it does not collect a complete

Table 2
Evaluated sampling points in the concentrating and pelletizing process.

Sample point	Primary sampling method	Primary increment interval	Analysis	Analysis interval
After primary milling	Manual grab sample	8 h	XRF	8 h
Before floatation	Slurry pressurized sampler	Part of the stream all of the time	XRF	20–30 min
Before floatation	Slurry pressurized sampler	Part of the stream all of the time	Specific surface area	3 min
Magnetite slurry	Slurry shark fin sampler	Part of the stream all of the time	XRF	20–30 min
Magnetite slurry	Slurry shark fin sampler	Grab sample from primary sample	Specific surface area	4 h
Waste	Slurry shark fin sampler	Part of the stream all of the time	XRF	24 h
Additives	Slurry shark fin sampler	Part of the stream all of the time	XRF	24 h
Slurry w. additives	Round pumping from slurry tank	Part of the stream all of the time	XRF	20–30 min
Filtered slurry	Manual grab sample	4 h	Moisture content	4 h
Final product	Cross stream sampler	5 min	Size distribution	1 h
			XRF	24 h
			Abrasion index	1.5 h
			Other physical and metallurgical assays	8 h/24 h

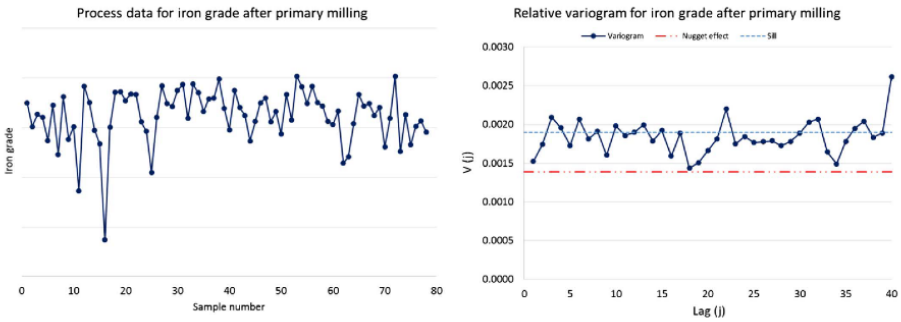

Fig. 2. Relative variogram for iron grade after primary milling in the concentrating plant. Upper dashed line indicating the sill and lower dash/dotted line indicating the nugget effect of the variogram.

cross section of the moving slurry stream, inducing a increment delimitation error (IDE) as well as a increment extraction error (IEE). As incorrect sampling errors are never constant (DS 3077, 2013, Minkkinen, 2004) this will influence the nugget effect of the variogram. In the present case, the nugget effect is however surprisingly small, indeed such a minor effect from a TOS incorrect shark fin sampler is rarely seen. The most likely explanation is that the material is significantly well mixed as it is ducted in pressurized pipelines.

For some time periods the variograms are exhibiting a clear periodicity of 8–10 h that is not as easily discerned in the raw process data. This periodicity can for example derive from fluctuations in milling capacity or overcorrections of the floatation, leading to episodic fluctuations which are not always distinguishable in the process data. Periodic fluctuations in the process are important to detect and counteract early, to avoid unnecessary process variability. The periodicity can also originate from the sampling system but this is just as important to identify and eliminate. Examples of actions that could lead to periodicity in the process are milling fluctuations, changes in crude ore, overcorrections in the control room and flushing (for cleaning purposes) of slurry sampling systems.

3.3. Moisture content of filtered slurry

A variogram for moisture content of filtered slurry is presented in Fig. 5. The sampling method for this analysis is manual grab sampling every 4 h, analysed at line using a rapid moisture analysis method. The sampling point is from the top of a conveyor belt load where several filters have discharged material at different stages, resulting in every subsequent filter loading material on top of the previous filter discharge. This leads to that the sampling mainly collects material from the last one or two filters rather than from the complete height of material on the belt. The variogram indicates a nugget effect to sill ratio of up to 60% across all evaluated variograms. This indicates that the sampling method does not have an acceptable precision.

3.3.1. Interpretation and improvements

The moisture sample is extracted using manual grab sampling leading to a high nugget effect to sill ratio. The sampling method collects material from the top of the conveyor belt and not from the complete cross section of material, which can never be representative. This means that the observable variation is lower than the actual variability present in the material from all filters. By applying alternative sampling methods based on TOS correct procedures, there are many possibilities to avoid incorrect sampling errors (ISE) and thus to lower the nugget to sill ratio, notably by employing composite sampling or a process analytical technology (PAT) sensor solution.

3.4. Size distribution of final product

A variogram for size distribution analysis results for 9.0–12.5 mm

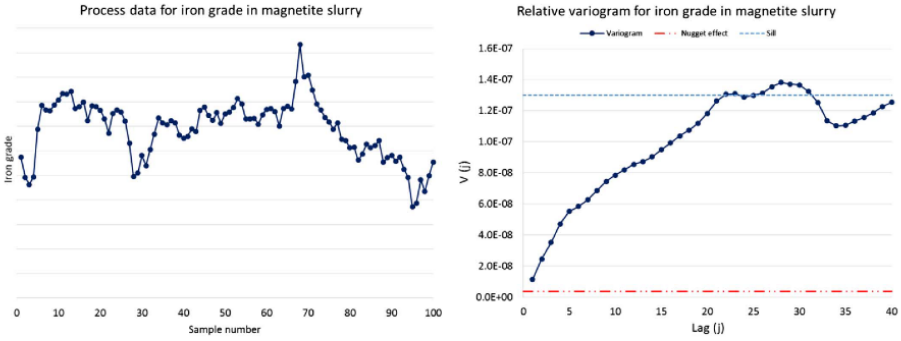

Fig. 3. Relative variogram for iron grade in magnetite slurry. Upper dashed line indicating the sill and lower dash/dotted line indicating the nugget effect of the variogram.

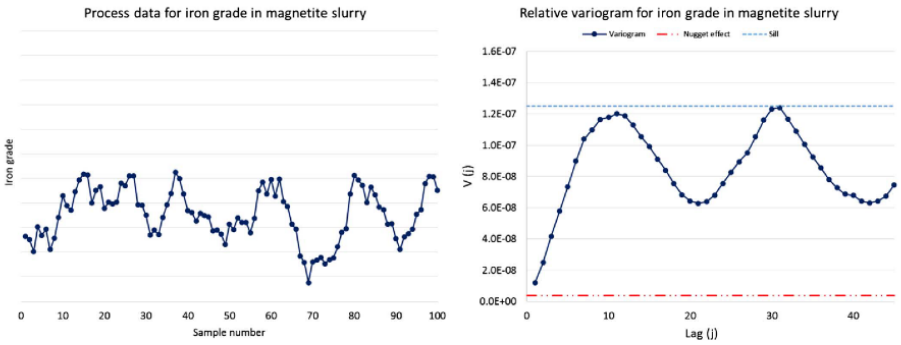

Fig. 4. Relative variogram for iron grade in magnetite slurry. Upper dashed line indicating the sill and lower dash/dotted line indicating the nugget effect of the variogram.

fraction is presented in Fig. 6. The sampling is an automated linear cross stream sampler. A primary sample is collected every five minutes and split for several analytical purposes. The size analysis sample is an aggregate of rotary division increments collected over one hour and analysed on-line by automated sieving. The variographic analysis of the specification size fraction of 9.0–12.5 mm shows a nugget effect to sill ratio of approximately 25–30% for all the evaluated variograms.

3.4.1. Interpretation and improvements

The sampling procedure is a state-of-the art automated linear sampler with an automated sieving analysis. The sampling system including analysis has been validated according to ISO 3085 and 3086 showing no sampling bias and a similar precision as the variogram presented here. The slightly elevated nugget to sill ratio for this measurement system is because the sill, i.e. overall process variation, is low in absolute terms for this stable process. Therefore there is no need to take action to improve the sampling procedure in this case.

4. Discussion and conclusions

The variographic characterization of sampling system is powerful to evaluate the performance of process sampling systems. Since process data is generated at regular time intervals the amount of data to evaluate is abundantly available on-line. This enables one to apply variographic characterization with regular intervals to evaluate and control the performance of sampling systems with any degree of resolution. If variographic analysis is continuously applied to process data, changes in process and sampling system variability can be easily detected and with early detection, and there is then a greater possibility to find quick solution to the problem at hand. To further evaluate the sources of variability collected in the nugget effect, methods like replication experiments (Esbensen and Wagner, 2016), gauge R&R studies (Montgomery, 2009) can be conducted. By applying these methods at different stages in the sampling and preparation system the short term variability or nugget effect can be decomposed into basic variance source components. To make continuous evaluation with variographic characterization possible, there is a need to automate the handling of process data for direct on-line application of appropriate variographic analysis. In this context there is a challenge in the form of reliable automated outlier detection and elimination (Esbensen et al., 2007). Identification and replacement of missing data points, generating uneven lag distances, need also to be "automated".

The sampling system evaluations presented in this paper range from TOS correct automated samplers to non-representative grab-sampling. As expected, the grab sampling methods show high nugget effect and large nugget to sill ratio, in contrast to the automated representative approaches. The diversity of sampling methods analysed here clearly indicates that sampling representativity has a strong manifestation through the nugget effect magnitude. TOS correct sampling systems will have a low contribution to the nugget effect. Any deviation in

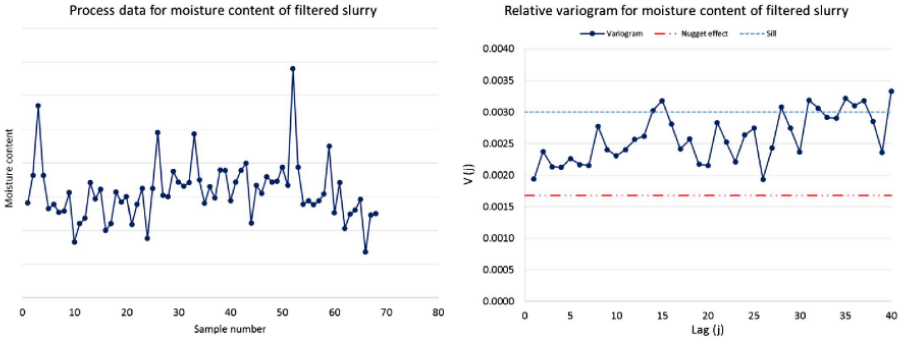

Fig. 5. Relative variogram for moisture content in filtered magnetite slurry. Upper dashed line indicating the sill and lower dash/dotted line indicating the nugget effect of the variogram.

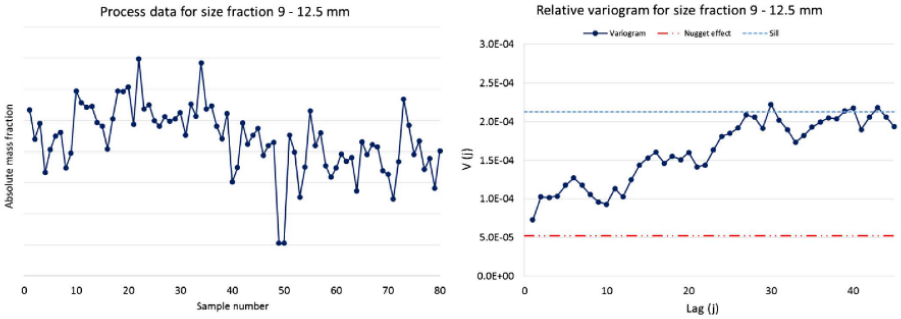

Fig. 6. Relative variogram for 9.0–12.5 mm size fraction of final product. Upper dashed line indicating the sill and lower dash/dotted line indicating the nugget effect of the variogram.

performance or variability in either sampling or process can be identified by applying variographic analysis at a regular basis.

Quality control methods in iron ore processing are often lacking assessment of sampling system performance, which often rely solely on visual inspections. Regular application of variographic analysis would remedy this issue. This would serve as a complement to regular statistical process control and laboratory QA/QC programs. Evaluation with the combination of these three control methods will achieve faster problem solving of deviating process control data, by giving a clear picture of where the problem has occurred: in the process, in the sampling system or in the laboratory. To successfully implement this combined quality control to all parts of the production, sampling and laboratory systems, good communication between process engineers, quality control engineers and laboratory engineers are essential.

4.1. Conclusions

Application of variographic analysis to process data in LKAB iron ore concentrating and pelletizing enabled characterization of sampling systems through the entire value chain (not all sampling system evaluations are shown here). Sampling and analysis variability components were quantified and opportunities for possible improvements of sampling systems was identified. Important true process variations and periodicities could be identified. Variographics could successfully decompose the total variability observed, as either stemming from the process or from imperfections in the sampling system. An interesting conclusion from the present study is that empirical variographic characterization is always necessary; one example actually transgressed conventional wisdom in that a shark fin sampler was found to have an acceptable nugget to sill ratio. However, we are well aware of the danger of generalizing such singular findings.

Extensive access to time series process data makes it possible to apply variographic characterization regularly, providing up to date control of process and sampling variability with any desired frequency. This would make specific experiments for precision evaluation of sampling systems redundant, resulting in substantial savings in money and resources (Thisted and Esbensen, 2017). At the same time, periodicity and small changes in process variation can be detected and counteracted early, minimizing the risk for producing products out of specification.

When variographic analysis is applied to process data at a regular basis, one obtains process variability characterisation as well as an evaluation the specific sampling and measurement system in use. The current study strongly supports previous suggestions that significant benefits can be obtained when applying variographic analysis to continuous industrial processes.

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Paper VI

Improvement of sampling for moisture content analysis in iron ore pellet feed – Variographic characterization and on-line IR-analysis

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IMPROVEMENT OF SAMPLING FOR MOISTURE CONTENT ANALYSIS IN IRON ORE PELLET FEED – VARIOGRAPHIC CHARACTERISATION AND ON-LINE IR-ANALYSIS

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ABSTRACT

Accurate information of physical, chemical and metallurgical quality characteristics such as moisture content, iron grade and particle size are crucial for successful process control and for producing products within customer specifications. Correct moisture content of pellet feed is an important variable for successful balling of iron ore pellets and to minimize power consumption in the sintering process. Control of the filtering process in the pelletizing plant is therefore essential for both productivity and high product quality. The current measurement systems for moisture content at LKAB are based on two types of manual grab sampling, subsequently analyzed with rapid moisture determination methods. Previous studies of the measurement systems have indicated high sampling variability resulting in lowered possibilities for successful process control. The objective of the present study is to evaluate an alternative filter sampling approach and testing of on-line Infra-Red analysis (IR-analysis). On-line IR-analysis has the advantage of eliminating physical sample extraction and allowing for continuous monitoring of moisture content. However, this approach is sensitive to the surrounding environment, making careful calibration and control crucial for the quality of measurement results. Furthermore, as IR-sensors only target the surface of the conveyed pellet feed, several *incorrect sampling errors* need to be considered and counteracted to eliminate sampling bias and reduce the remaining measurement variability. Variographic characterization was applied to the two current grab sampling approaches of combined filtered material, to the alternative filter sampling method, and to the On-line IR-analysis. The results indicate that both the sampling of separate filters (using composite sampling and ISO-standardized moisture analysis), as well as a calibrated and well-maintained IR-sensor can improve the relative variability significantly, by lowering the total measurement system variance from 60% to 25%.

KEYWORDS

Moisture content, iron ore pellet feed, sampling, on-line, variographic characterization

INTRODUCTION

Luossavaara Kiirunavaara limited company (LKAB) is an iron ore mining company located in the north of Sweden. LKAB is currently operating several iron ore mines, concentrating and pelletizing plants. The largest mine is the Kiirunavaara apatite magnetite deposit, mined under-ground with the main level at 1365m below ground. LKAB's core business is producing high quality iron ore pellets for blast furnace and direct reduction steel making. The customer requirements for size, chemical content as well as several metallurgical parameters of finished pellets are strict, demanding extensive and precise process control through the complete value chain, from the mine to refining and pelletizing plants.

For most industrial production processes, monitoring and control of key process and quality parameters is crucial. In the pelletizing process of LKAB, moisture content of filtered iron ore pellet concentrate (KPC) is an important parameter for successful balling of pellets. The moisture content needs to be correct to achieve correct pellet size and strength, and to maximize produced tonnage (Tomifeeva et al., 2015). Furthermore, a more precise moisture determination and control of the filtering process could enable lower moisture variability in the raw pellets and thereby a lowered power consumption for sintering. Simultaneously, moisture sampling is a complicated task and representative samples are not always achieved (Ziegelaar and Fritz, 2017). In process monitoring and control, it is important to consider the total measurement variability when determining how and when to react to process variability as observed in process control charts. For many measurement systems, the analytical uncertainty is well determined and controlled, while the primary sampling is conducted according to traditional protocols, not taking Theory of Sampling (TOS), or sampling variability into account (Minnitt and Esbensen, 2017). Compliance to TOS, DS 3077 (2013), and the international standard for sampling of iron ore (ISO 3082, 2009) have improved in recent years, especially for commercial purposes (Holmes, 2017). Regardless, poor sampling practices are still frequent in process control practices, even though the use of TOS-correct sampling practices are critical to allow for effective control of important process parameters. Moisture samples are especially important to handle correctly so as not to induce sampling and preparation bias due to evaporation or contamination during the sampling process (Holmes, 2016).

The current protocol for moisture determination at LKAB plant 1 is a manual grab sample taken from the top of a conveyor belt, *belt sampling*, transgressing several TOS principles. The

complete primary sample is analyzed using a rapid moisture method. The conveyed material is discharged from five consecutive filters, meaning that KPC from the first filter is discharged on the bottom of the conveyor belt and KPC from the last filter is discharged on top of the belt. Previous studies of this sampling system have indicated a high variability and a high nugget-to-sill ratio, resulting in lowered possibilities for successful process control (Engström and Esbensen, 2017). This has initiated evaluations of alternative sampling methods as well as a possible on-line moisture analysis solution. The current sampling system for LKAB plant 2 is based on *spear sampling*, extracting increments as a complete column from the top to bottom of a conveyor belt. Three primary increments are combined to a composite sample and analyzed using a rapid moisture method. The sampling location is situated shortly after a belt reloading point, expected to achieve some mixing of the material discharged from the previous belt.

The objective of the present study is to evaluate, and if possible, improve the sampling variability for the total measurement system for moisture determination of KPC in LKAB pellet plants. The study will compare the current sampling methods to the alternative filter sampling approach, Table 1. Furthermore, an on-line IR-analysis sensor will be installed on top of the conveyer belt in plant 2 and assessed in comparison with the manual sampling methods.

Table 1 - Descriptions of the evaluated sampling systems.

Sampling method	Description	Plant	Evaluation method
Belt sampling (current)	Manual grab sample extracted from the top of a conveyor belt holding filtered material from five consecutive filters.	1	Variographic characterization of process data. Variographic experiment
Filter sampling (new)	Manual extraction of a three-increment composite sample. Increments are extracted from each filter as the material is discharged.	1	Variographic characterization of process data. Variographic experiment
Spear sampling (current)	Manual extraction of a three-increment composite sample. Increments are collected using a spear, extracting a complete column from the top to the bottom of a conveyor belt holding filtered material from all filters.	2	Variographic characterization of process data.
IR-analysis (new)	IR-sensor installed on a plow, on top of the conveyor belt close to the spear sampling location.	2	Variographic characterization of process data.

MATERIAL AND METHODS

The Fundamental Sampling Principle (FSP) of TOS is that all particles of the lot being sampled should have equal probability of being sampled and subsampled to become a part of the final analytical aliquot (Gy 1979; Pitard 1993; DS 2013). It is critical to ensure that all samples are representative, i.e. free from bias and with acceptable reproducibility (precision), in order to be able to

control the target process parameter accurately. Moisture content can change during sampling and sampling preparation, for example due to evaporation, surrounding moisture and other contamination sources (Ziegelaar and Fritz, 2017). This is one reason for the current manual sampling systems used for moisture determination of KPC. The manual sampling extraction and rapid moisture analysis methods are designed to achieve a short time between sampling and analysis, minimizing the risk for external factors affecting sample integrity. However, the current measurement systems have been questioned by process engineers, as they do not visualize the true process variability in a satisfactory fashion, likely due to non-representative samples. The current manual sampling methods fail to collect the complete cross section of the one-dimensional material stream and are thereby violating the FSP. This is likely leading to significant IDE and IEE that generate sampling bias (Gy 1979).

Filter Sampling

An alternative sampling method, *filter sampling*, has been suggested to improve sampling variability and enable more precise process control in plant 1. Filter sampling is a manual method, extracting one composite sample from each separate filter. The primary increments are collected from the falling material as it is discharged from the filter on to a conveyor belt. Three increments are collected from the middle and each side of the filter and combined in to one composite sample of approximately two kilos per filter. The full composite is analyzed using conventional drying according to the standardized method for moisture determination of iron ore, ISO 3087 (2011). The response time for the standardized moisture determination (ISO 3087, 2011) is minimum five hours, compared to the current rapid moisture method generating results in approximately 20 minutes.

The filter sampling method collects a three-increment composite sample, and the precision is expected to improve compared to the belt sampling that only extract one increment/primary sample (Minkkinen and Esbensen, 2009). As this method produce one analytical result for each filter, rather than one combined result for five filters, it will lead to a larger number of samples, as well as longer time for analysis. The incentive for the increase in time and resources is to enable more specific control of each filter and thereby improve the product quality. In comparison, the current belt sampling method only generates one moisture content result for the combined material from all five filters, making it difficult to use process control data only, to discern which filter needs to be adjusted. Control and adjustments of individual filters is today largely dependent on visual inspection and operator knowledge. With a more precise measurement system; moisture variability and thereby power consumption in the sintering process could be lowered. Possibilities for future automation of the filtering process is also dependent on a more precise measurement system for individual filters.

On-line Infra-Red Moisture Analysis

On-line analysis can produce rapid and continuous measurements, often enabling significant improvements in process control. Real time data give immediate feedback to upstream process control, allowing faster reactions to deviating results (Kurth, 2016). However, correct measurement results are dependent on the analyzed material being representative for the lot and that the measurement system is calibrated and validated correctly. On-line measurements are mainly applicable for process control and does not necessarily substitute all sampling. However, as the turn-around time for analytical results is lowered, more rapid and precise process control can often be achieved (Kurth, 2016). On-line sensors eliminate the demanding and error generating exercise of extracting and preparing physical samples as a routine protocol. The sampling error potential is instead transferred to the sensor, meaning that the sensor needs to analyze a representative part of the 1-D moving lot to be able to generate valid measurement results. It is often unrecognized that there is a complete duality regarding incorrect sampling errors between physical sample extraction and sensor analysis techniques (Esbensen and Paasch-Mortensen, 2010). IR-analysis is one commonly used technique for moisture determination of particulate material in the industry today (Cancilla, et al. 2003). However, IR-sensors are only able to do a surface determination, leading to difficulties in ensuring representativity of the measurement, due to the incorrect sampling errors IDE and IEE arising from not "sampling" the complete depth of the moving lot. Furthermore, IR-analysis is affected by the optical absorption of the material, distance to the material surface and surrounding light, temperature, dust and water vapor. This mean that calibration, validation and regular performance control of the IR-sensor is crucial for correct measurements.

An on-line IR-sensor was installed in LKAB plant 2. As the sampling location in plant 2 is situated immediately after a 90-degree belt transfer point, some mixing of material is assumed and the segregation between layers is believed to be lowered. The degree to which these compensating procedures helps has not been studied in detail. The IR-sensor is installed with an attached plow, lying on top of the material stream. The purpose is to create an even surface for measurements and to continuously adjust the height of the sensor so the distance to the analyzed material is consistent. The IR-analysis only enables moisture determination of KPC from all filters simultaneously and thereby serves as a general control of the produced material. However, no information regarding separate filters can be discerned and further control is needed to pinpoint any problems stemming from individual filters.

Variographic Characterization

Variographic analysis is a powerful tool for evaluation of auto-correlated process data. The variogram characterizes the heterogeneity in a process stream by analyzing consecutive increments or samples (Pitard, 1993, Esbensen et. al. 2007, Minnitt and Esbensen, 2017). With systematic use of

variograms of routine process data, true process variability can be decoupled from the total measurement system variability, including sampling (Esbensen and Romañach, 2015). Variograms have three characteristic features used for interpretation of both sampling and process variation; the range, the sill and the nugget effect. The sill is the maximum variation within the analyzed data. The sill is the level where the variogram has leveled off and beyond this point no auto-correlation exists between samples. The range is the lag distance where the variogram reaches the sill, meaning that samples taken at lag distances above the range are not auto-correlated (Esbensen et al., 2007; Minnitt and Esbensen, 2017). The nugget effect is calculated by back-extrapolation of the variogram to lag zero. The intercept with the Y-axis of the variogram indicates the fictive situation of two samples collected at the same instance or physical location. The nugget effect is comprised by all errors that characterize the total measurement system, including sampling and analysis, and can be interpreted as the minimum possible error. (Esbensen et al., 2007). Another important parameter of the relative variogram is the *nugget-to-sill ratio*. This ratio is an estimation of the percentage of total variability observable in the data which does not stem from the process variation, but solely from the total measurement system (primary sampling/splitting/analysis). By assessing the nugget-to-sill ratio, the quality of the measurement system can be gauged and compared to alternative sampling systems under evaluation. A recent introduction to this feature was presented by Esbensen and Romañach (2015). The full mathematical description for variographic analysis, as well as quantitative estimation methods for the inherent variability contributions, are presented in full detail in historical TOS literature and in recent publications (e.g. Gy, 1979; Pitard, 1993; Esbensen and Paasch-Mortensen 2010; Minnitt and Esbensen 2017).

Relative variograms are used for comparison between the measurement variabilities of the separate measurements systems in this study. Variographic analysis was applied to a plethora of available long-term process control data from plant 1 and 2, for comparison and evaluation of the current belt sampling and spear sampling systems *vs.* the installed on-line IR-analysis. Variographic experiments were also executed in plant 1, both to the current belt sampling method and the alternative filter sampling method. The objective of the experiments was to evaluate both sampling methods in more detail, by collecting and analyzing samples more frequently then what is done for routine process control.

RESULTS AND DISCUSSION

The results of this study are a combination of qualitative assessment and variographic data analysis. The qualitative assessments were performed by process and research engineers working with the evaluated measurement systems and process control on a day-to-day basis. The variographic characterizations were applied to both long term process data as well as to the specific variographic experiments performed. Several variograms representing different time periods were also analyzed for

the long-term process data. As the general appearance of the variograms were similar for all time periods, only one variogram for each sampling method is presented here.

Filter Sampling Method

The filter sampling method was tested in a six-month campaign, extracting one sample every 24 hours, Monday to Friday, from all filters in production in plant 1. As the time between analytical results (the lag) in this campaign was not constant (no samples were extracted Saturday and Sunday), variographic analysis cannot be applied to these data. However, qualitative analysis of the filter sampling data indicates that the total variability within each filter is equal, or lower than for the current belt sampling method. Short time periods with large variations are present but can be deduced to originate from filter screen changes, rather than true process variations.

The variographic experiment for the belt sampling method resulted in 48 samples extracted during 12 hours and the variographic experiment for the filter sampling method resulted in 66 samples extracted during 22 hours. Variograms for both variographic experiments as well as a variogram for the belt sampling process data are compared, Figure 1. Belt sampling is indicating a nugget-to-sill ratio of approximately 60% for the long-term process data. For the shorter variographic experiment, the nugget effect is lower, but the variogram is flat, indicating that the sampling variability covers all existing process variability. The high nugget-to-sill ratio indicates that this measurement system is not suitable for process control, as more than half of the variability in the data is stemming from the measurement system, leaving only a small fraction as true process variability. One contributing factor to the high nugget-to-sill ratio may be the incorrect sampling errors, IEE and IDE, present in the belt sampling method. These errors are a direct consequence of the incorrect sample extraction, as the method is not collecting a complete cross section of the material stream.

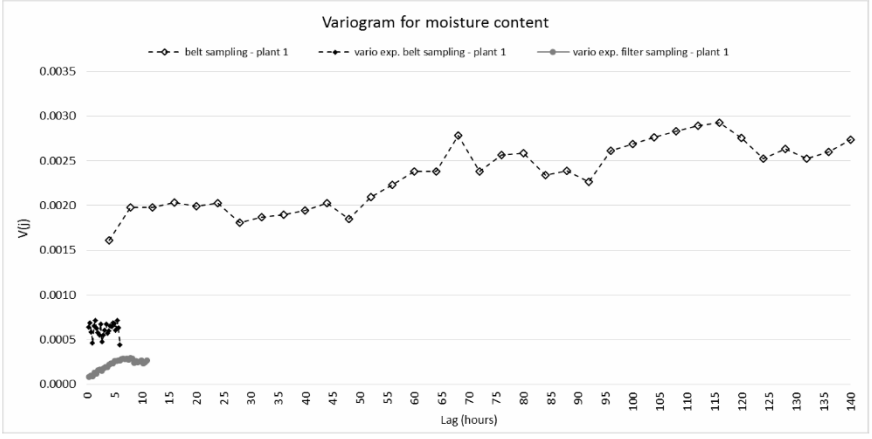

Figure 1 - Relative variograms of belt sampling for long term process data as well as for variographic experiments of the belt sampling and filter sampling methods in plant 1.

The variogram for the filter sampling method shows a nugget-to-sill ratio of approximately 25%, a significantly lower sampling variability than for the belt sampling method, Figure 1. This shows that the filter sampling method could enable improvements for process control of the moisture content in KPC. Even though the filter sampling approach is not collecting the complete cross section of the material discharged from each filter, the three increments are covering both the with and depth of the material. As the nugget-to-sill ratio is acceptable, any present sampling bias does not seem to affect the sampling method unduly. To manually collect a complete cross section of the discharged filter material is impossible due to the large sample mass this would involve. However, as the manual filter sampling method is showing positive results regarding sample precision, reflections on further development of automated sample extraction of the complete cross section of the filter discharge are in progress. The filter sampling method results in five times as many samples as the belt sampling method and leads to longer response time for analysis. However, as the measurement variability for the filter sampling is significantly lowered and the process variation for individual filters is low, the time between samples could possibly be lowered compared to the current belt sampling method. The longer response time for analytical results is accepted by process engineers and operators, as the results have better precision and are more reliable than for the belt sampling method.

On-line IR-Analysis

The on-line IR-analysis is installed in plant 2 and have been tested for several months. Problems with the calibration of the IR-sensor have resulted in several time periods where the sensor and the manual samples show large discrepancies. Therefore, only time periods with stable and correct calibration have been used for the variographic analysis of the IR moisture measurements. The variogram for the on-line IR-analysis is compared to both the current spear sampling in plant 2, as well as to the belt and filter sampling methods in plant 1, Figure 2. The variograms show that the IR-analysis, the spear sampling and the filter sampling have similar nugget effects and similar sills. The nugget to sill ratio is approximately 25 % for all three methods and are thereby significantly lower than for the belt sampling method used in plant 1. Similar variogram appearances indicate that the methods perform equally well regarding precision. However, the IR-analysis presents real-time results every 10 minutes, which is an advantage compared to spear sampling that is routinely performed every two hours and the analysis takes about one hour before the drying is finished and results can be presented. The On-line IR-analysis could therefore allow for significantly faster detection of important process changes. This could lead to improvements of the process control systems and possibly enable future automation. The IR-analysis for moisture determination could also be applied to other parts of the refining process, if proven representative and precise.

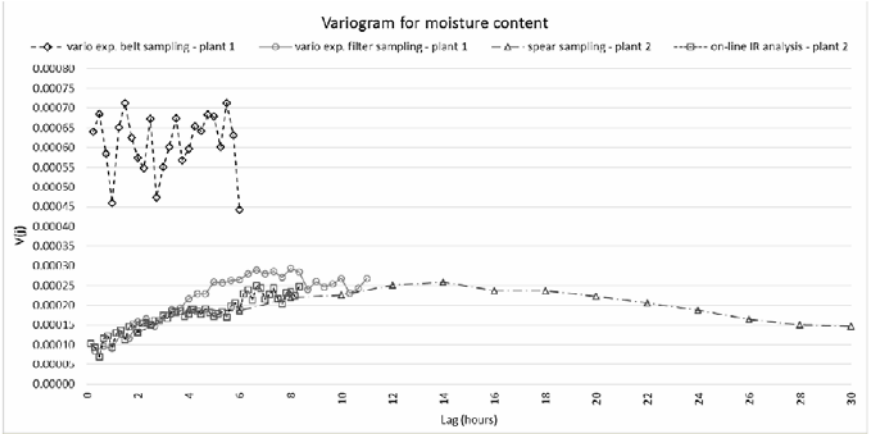

Figure 2 - Relative variograms for belt sampling and filter sampling data from variographic experiments and for long-term process data from spear sampling and on-line IR-analysis.

As the relative measurement variability for the IR-analysis is similar to both the spear sampling and filter sampling methods, the main advantage of the IR-analysis is the rapid response, rather than increased sampling precision. However, the non-trivial calibration problems encountered need to be handled before the method can be readily applied for process control. Furthermore, the current application of the IR-sensor only gives one overall moisture measurement and does not reveal the performance of separate filters, which is a disadvantage compared to the filter sampling method. The qualitative evaluation of the IR-sensor results indicate that the calibration problems could be associated with the varying number of filters in production. This is causing changes in production rate, leading to significant variations in tonnages on the belt, which in turn lead to height and compaction variations in the material that the current plow setup cannot eliminate satisfactorily. The incorrect sampling error challenges are being worked on at this time; TOS in combination with creative engineering solutions are applied to find means to solve these challenges. Further testing and evaluations of the IR-sensor is needed to find a solution to the calibration problems and allow for a stable measurement framework that is not affected by changes in production rate or product quality.

CONCLUSIONS

This study concludes that the current belt sampling method causes sampling bias and high measurement variability due to the violation of FSP, leading to the incorrect sampling errors IDE and IEE. The variographic characterization shows that the sampling variability is high, and the nugget-to-sill ratio is approximately 60%. The alternative filter sampling method exhibits a significantly lower sampling variability than the belt sampling method. The nugget-to-sill ratio of the filter sampling

method is approximately 25%, indicating that the method is *fit-for-purpose* for control of the filtering process (DS 2013). The larger variations visible in the filter sampling data, for isolated time periods, are mainly due to filter cloth changes rather than true process variability. During normal production, the overall variability is similar or lower than the belt sampling method. The performance and variability information of each filter is particularly useful for successful adjustments of each individual filter, after filter cloth changes. The filter sampling method is also able to detect unexpected shifts or problems in individual filters. These are important advantages compared to all the other evaluated methods that cannot discern the moisture content stemming from each individual filter.

The overall conclusion of this study is that dedicated filter sampling is the preferable method as it leads to significantly lower measurement system variability, compared to the belt sampling method. The filter sampling also enables process control of individual filters, which is not possible with any of the other evaluated methods. The IR-analysis need further work to eliminate first deployment calibration problems and be able to handle changes in the production rate. Further studies could include automation of the filter sampling method, and evaluation of IR-analysis applied to each filter separately, to achieve the advantage of real time analysis as well as process control of individual filters.

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Paper VII

Total process and measurement system characterization using variography – covering the complete ore-to-shipping value chain at LKAB

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Variographic Assessment of Total Process Measurement System Performance for a Complete Ore-to-Shipping Value Chain

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Abstract: Variographic characterisation has been shown to be a powerful tool to assess the performance of process measurement systems, using existing process data. Variogram interpretation enables decomposition of variabilities stemming from the process and measurement system, respectively, allowing to determine if measurements are able to describe the true process variability with sufficient resolution. This study evaluated 14 critical sampling locations, covering a total of 34 separate measurement systems, along the full processing value chain at Luossavaara Kiirunavaara limited company (LKAB), Sweden. A majority of the variograms show low sill levels, indicating that many sub-processes are well controlled. Many also show low nugget effect, indicating satisfactory measurement systems. However, some notable exceptions were observed, pointing to systems in the need of improvement. Even if some of these were previously recognized internally at LKAB, the use of variographic characterisation provide objective and numerical evidence of measurement system performance. The study also showed some unexpected results, for example that slurry shark-fin and spear sampling show acceptable variogram characteristics for the present materials, despite the associated incorrect sampling errors. On the other hand, the results support previous conclusions indicating that manual sampling and cross belt hammer samplers are leading to unacceptably large sampling errors and should be abandoned. Such specific findings underline the strength of comprehensive empirical studies. Based on the present compilation of results, it is possible to conduct rational enquiry of all evaluated measurement systems, enabling objective prioritization of where improvement efforts will have the largest cost–benefit effect.

Keywords: iron ore; process sampling; variographic characterisation; Theory of Sampling (TOS); theory of sampling; measurement systems; quality control

1. Introduction

Grade control is an essential element in quality assurance and quality control (QA/QC) of refined and high-quality products in many global process industry sectors, e.g., in fields from mining, ore and minerals processing to cement, pharmaceuticals, food and feed. Grade control is used to assess the quality of intermediary and final products and to control and adjust a range of critical process parameters. Grade control can be conducted in a variety of fashions specific for different situations e.g., on-line measurements, sensor measurements, in-line or at-line sampling and measurement

systems, automatic or manual sampling. However, a key factor in all methods for grade control is sampling. No matter if the measurements include physical sampling extraction, or pointing a sensor to a process stream, sampling is being conducted as analysis is only addressing a very small part of the lot (physical extracted sample) or of a stream (the part interacting with the sensor). All sampling processes generate a complement of sampling errors that only can be eliminated or minimized through understanding of how and why they originate [1–3]. The Theory of Sampling (TOS) presents a complete framework for how to eliminate the so-called 'incorrect sampling errors' (ISE) and evaluate the magnitude of the remaining error effects called 'correct sampling errors' (CSE). The representativity of an extracted sample is of utmost importance in order to optimize resource utilisation, maximize profitability and minimizing financial risk in the complete process value chain [4]. The rationale for representative sampling, the Fundamental Sampling Principle (FSP), is that all lot units (grains, fragments, increments) have equal opportunity of ending up in the final sample. A representative sample is both accurate and reproducible, i.e., ISE have been eliminated, and CSE have been minimized through appropriate sampling procedures [2,5].

This paper gives a brief background regarding TOS and variographic characterization and presents an overview of all evaluated measurement systems. It also includes in-depth discussion of specific variograms with deviating appearances (i.e., high or unexpected variability or periodicity). The complete TOS framework and terminology is presented in the sampling standard DS 3077 [6], as well as in the other references given above.

1.1. Luossavaara Kiirunavaara AB

Luossavaara Kiirunavaara limited company (LKAB) is a state-owned iron ore mining company located in the north of Sweden. LKAB was founded in 1890 and has supplied the world steel market with iron ore products and been an important cog in Sweden's industrial development for more than a hundred years. LKAB's competitive positioning is as a high quality, sustainable supplier of processed iron ore; the main product is iron ore pellets either for blast furnace (BF pellets), or for direct reduction (DR pellets) steel making. LKAB's iron ore operations include two underground mines and one open pit mine in Kiruna, Malmberget and Svappavaara in northern Sweden respectively. Sorting, concentrating and pelletizing plants at the three mine sites refine the mined magnetite ore to iron ore pellets, requiring only one third of the carbon emissions necessary for sintering compared to hematite pellet or one seventh compared to sintering of fines.

As the competitive advantage of LKAB is processed specialty iron ore products, and the aim is to deliver added value to customers through high quality products, it is critical to employ correct grade and quality control in all parts of the mine-to-product value chain. This is done through continuous sampling and analysis at all critical process steps, using both physical sampling combined with laboratory analysis, as well as various types of on-line, in-line and at-line measurement systems. Important quality characteristics evaluated in the grade control in LKAB processing plants are, e.g., size distribution, iron grade, silica grade, phosphorus grade, moisture content, specific surface area, crushing strength, abrasion index.

1.2. Variographic Characterization of Measurement Systems in Process Industries

Control of the LKAB production process is based on a variety of measurement systems, and the possibility for effective and correct process control is critically dependent upon representative samples. Process variability is the key monitoring objective, and the true process variability cannot be allowed to be dampened by variability components stemming from the measurement system. The total process data uncertainty is the sum of error contributions stemming from sampling, sub-sampling, sample pre-processing and analysis. Variographic characterization of continuous process data is a powerful tool for comprehensive understanding of the important relationship between process and measurement system variability [7,8], in effect providing a quality control tool for total process measurement systems. The different variability sources in process data are not identifiable using

traditional statistical process control (SPC), while variographic characterization allow for decomposition into individual contributions.

Several publications exemplify and advocate the use of variographic characterization for evaluation and improvement of process measurement systems for a large variety of processes e.g., [7–14]. Variographic characterization is effective for evaluation of (i) measurement system performance [9], (ii) comparison between different measurement systems [15] and (iii) detection of adverse process variations [16,17]. These recent publications point to a broad spectrum of information that can be retrieved by applying variographic characterization to historical and current on-line process data. It is also a favourable option to conduct a variographic experiment in which to obtain information by perturbing existing processes where- or whenever this is possible.

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1.3. Study Objectives

In contrast to many published case studies, focusing on one specific sampling system or part of the full process, this study is taking variographic characterization one step further, by applying it throughout the entire ore processing value chain in the LKAB processing plants in Kiruna, Figure 1. The aim is specifically to detect objective patterns for the range of process measurement systems deployed, and to decouple the measurement variability from the true process variability. By applying the same kind of variographic analysis to 'all' measurement systems and parameters used for process and quality control, in the sorting, concentrating and pelletizing processing plants, this study aims to compare measurement system performance across the entire mine-to-product pathway. This survey will then enable focused attention to the systems in most need of improvement. An additional objective is to detect patterns regarding the suite of different sampling methods, sampling intervals or analytical techniques used. This kind of complete plant process measurement system characterisation has not been performed before at LKAB.

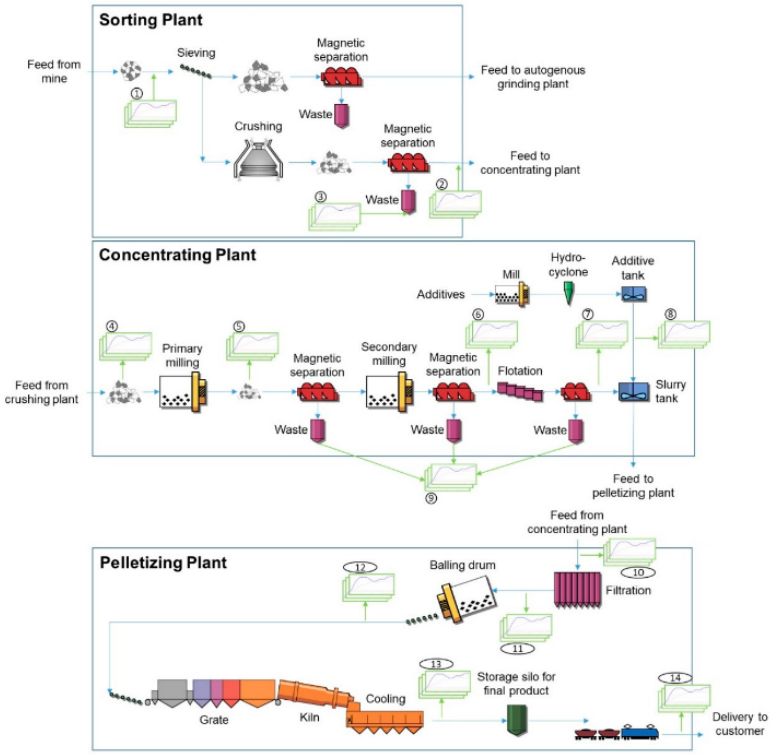


Figure 1. Schematic overview of the processing value chain at LKAB, Kiruna. Numbers (in circles) indicate the 14 critical sampling locations where variographic characterization of measurement system performance have been applied.

2. Theory of Sampling (TOS)

The first vestiges of what came to be the Theory of Sampling (TOS) was initially described in 1950 and was subsequently developed over a period of 25 years Pierre Gy. Today TOS ranges as the only complete and comprehensive theory covering sampling of particulate materials [3], and many other types of heterogeneous materials. TOS include practical sampling unit operations, systematization of eight sampling errors and heterogeneity characterization. TOS is comprised by all necessary concepts and process descriptions needed to reach the overall aim of representative sampling [1–3,6,18].

Representative samples are imperative for valid process monitoring. It is not possible to ascertain the status of an individual sample from any characteristic pertaining to the sample itself; it is only possible to guarantee representativity of the sampling process. The role of TOS is to supply all tools necessary to always be able to design and perform representative sampling processes.

2.1. Lot Heterogeneity

All sampling operations are error-generating processes; sampling errors are a direct consequence of the interaction between the sampling process (sampling method and the equipment used) and

the heterogeneity of the material. Lots of particulate materials have an inherent heterogeneity that can be discriminated at two scale levels, i.e., constitutional heterogeneity (CH) and distributional heterogeneity (DH). CH is the expression of the compositional differences between individual fragments (grains) in the lot at its current state of comminution, meaning that mixing or homogenization does not affect the CH. A sample extracted from a lot is among other affected by an error related to the CH, defined as the Fundamental Sampling Error (FSE) [3].

DH is the heterogeneity at a larger scale level between the mass of the increment and the full lot. DH denotes the compositional difference between increments (group of neighbouring fragments) of the lot. CH and DH are closely related; indeed, DH is a fraction of CH of the material being sampled. However, DH is also dependent upon the grouping factor, i.e., the mass of the sampled increment, and the segregation factor, i.e., the degree of structural heterogeneity of the distribution of the particles [2]. DH is the primary concern for the process of sample extraction as practical sampling is carried out by extracting increments from the lot. DH is a reflection of the spatial heterogeneity in the lot and the error generated by DH is termed the grouping and segregation error (GSE).

If DH were nil (an ideal state of complete spatial homogeneity), GSE would be zero, leaving only FSE. If the lot material were homogenous (an ideal, unreachable state for all materials), FSE would also be zero. However, sampling of all realistic materials in science, technology and industry, will always have some vestige of remaining GSE irrespective of how much this has been suppressed (often GSE is only suppressed to a level so that GSE + FSE can be proved to fall below an a priori expressed maximum acceptance level). The logistics of practical sampling, to reach a state in which FSE + GSE are the only effective sampling errors remaining, a correct sampling process, is an essential part of TOS. To this goal practical representative sampling is always first about how to eliminate the *incorrect* sampling errors [1–3,6,18].

2.2. Process (1D) Sampling and Sampling Errors

The heterogeneity carried by the full lot can be described as a complex totality of CH and DH, where the dimension of the lot is an important determinant. Lot dimensionality is related to the effective number of dimensions involved in physical extraction of a sample form the lot. Zero dimensional (0D) lots, i.e., all smaller batches which can be mixed, manipulated, moved and sampled with complete correctness, are only affected by the heterogeneities described above. Regarding industrial processes, as the ones addressed in the present study, the one-dimensional (1D) lot is the most common lot configuration. 1D lots are defined as elongated piles or process streams of material that can be sampled correctly through an increment-taking process, where a full (and intact) cross section of the material is extracted at regular intervals [19].

In process sampling, increments are often combined (aggregated) to form composite samples. Process sampling is, apart from the short-range heterogeneity fluctuation error (CE_1), which is the sum of FSE and GSE, also affected by the long-range fluctuation error (CE_2), called the trend error, and the periodic fluctuation error (CE_3) [3]. These two errors are not solely dependent upon the properties of the material in the lot, but also on variations stemming from the production processes and/or operator actions.

The process of representative sampling starts with elimination of all incorrect sampling errors (ISE), related to wrongful materialization of the primary increments. ISE includes the increment delimitation error (IDE) related to how the equipment for sample extraction is designed, the increment extraction error (IEE) related to how the sample extraction is performed, the increment weighing error (IWE) related to the weighing of individual increments and the increment preparation error (IPE) representing all adverse changes to the sample after extraction [3]. To the degree that these errors have not been fully eliminated, this will generate a sampling bias. In contrast to the ISE, the correct sampling errors (CSE), comprised of FSE and GSE, cannot be fully eliminated. However, to achieve representative sampling with acceptable precision, the CSE need to be satisfactorily minimised through appropriate sampling protocols [6]. Reduction of GSE is achieved by extensive mixing or blending

of the lot, and/or by increasing the number of primary increments making up a composite sample. The most straightforward way of reducing FSE is to comminute the lot material to a smaller particle size and/or increase the sample mass, of which the former is by far the most effective [6].

The sampling bias is by nature inconstant. In contrast to an analytical bias, the sampling bias cannot be statistically quantified and can therefore not be corrected for. The sampling bias is an unavoidable consequence of non-eliminated ISE. In practical situations, a sampling bias will inflate the total measurement system variability unnecessarily. This variability cannot be reduced by increasing the sample mass, number of increments, or replicating the analysis, it can only be eliminated by applying TOS correct sample extraction protocols.

Practical sampling: Practical sample extraction from industrial processes can be performed in countless different ways. Correct sampling processes are essential for valid process control decisions. Nevertheless, poor sampling practices are often found in the mining industry, leading to suboptimal use of resources, increased costs and loss of revenue [4]. The most important rule of process sampling (both slurries or dry particulate material) is to extract a complete cross section of the material stream at regular intervals. This is preferably done at a transfer point between conveyor belts or at dedicated pipeline sampling stations. In most practical mineral process situations, manual sampling is not sufficient as the flow rate generally is too high to enable extraction of a complete cross section. Cross belt cutters (i.e., hammer samplers), are not recommended as they are unable to materialise the complete depth of the ore stream without damaging the conveyor belt and therefore leaving a residual of fine material on the bottom of the belt [20]. A correctly designed cross stream sampler is the preferred solution for extracting representative process increment and samples. These can be combined with automated secondary sampling equipment (e.g., rotary sample dividers) and different forms of automated analytical equipment (e.g., size determination, crushing strength, moisture content or XRF) [21]. Slurry samples in mineral processing are often extracted from long and sometimes pressurized pipes. Common current sampling equipment include shark fin and spear samplers, but these are only extracting a part of the stream all of the time. As they do not result in a complete cross section, severe IDE and IEE are introduced, thereby generating a sampling bias [3]. A perfect TOS correct method for slurry sampling is the Vezin sampler operating on falling streams [22].

2.3. *Variographic Characterization*

To evaluate the performance of a process measurement system, it is important to understand all the different sources of variabilities. These include (i) sampling error contributions, (ii) analytical errors and (iii) true process variations. There can be large variations in the relative proportions of these error components and their uncertainty influences on any given process data series. In principle, each process situation needs to be assessed on its own merits, which is where TOS' general principles come to the fore.

Variographic characterization is a structured way of estimating the magnitude and origin of different sources of variability in process data, including estimating the so-called minimum practical error (MPE), aka the nugget effect (V(0)) [1]. Variographic characterization is applicable to all ordered 1-D time or space data series, e.g., process monitoring data or similar experimental data. The variogram most often used is characterized by the relative heterogeneity contributions (h_q), equation 1, rather than the absolute analytical concertation (a_q). The relative variogram is applied in this study as the focus is to compare variograms for the different sampling protocols in use at LKAB.

The method of variographic characterization, including full mathematical description and all evaluation criteria, is presented in detail in the historical TOS literature and in more recent overview publications e.g., [1–3,5,7,8,11]. Point calculations of the relative semi-variogram is following

Equations (1) and (2). This variogram is often called the semi-variogram, because of division by a factor 2 needed in order to express the variability as a standard statistical variance.

$$h_q = \left(\frac{a_q - a_L}{a_L} \right) \frac{M_{s_q}}{M_s}. \quad (1)$$

$$v(j) = \frac{1}{2(Q-j)} \sum_{q=1}^{Q-j} (h_{q+j} - h_q)^2, \; j = 1, 2, \ldots, \frac{Q}{2}. \quad (2)$$

$v(j)$: variogram point estimations (expressed as statistical variances)
Q: total number of measurements used for variographic characterisation
j: lag (distance *between* samples in time or space)
h_q: heterogeneity contribution for the sample q
a_L: mean analytical concentration for the lot
a_q: analytical concentration for sample q
M_s: sample mass (or increment mass).

There are three main characteristics of a variogram: the range, the sill and the nugget effect, all explained in Table 1. TOS is the theoretical framework that has analysed the full meaning of the nugget effect in process sampling, by specifying it as the sum of all remaining ISE plus all CSE, as well as the Total Analytical Error (TAE) effects associated with the current sampling protocol. The nugget effect (or MPE), is thus an estimate of the sum of all residual variance contributions from the total measurement system (sampling, preparation and analysis). Table 1 presents a brief description of the interpretation of the individual components in the semi-variogram.

A critically important feature of the semi-variogram is the nugget-to-sill ratio. This ratio expresses the relative percentage of the variance that is related to the total measurement system in comparison to the overall observable variability, represented by the sill [2,3,23]. This ratio is a quality grading of the total process measurement system; it must be smaller than 30% in order for the process measurement system to be qualified [6]; for specific applications a lower threshold may be appropriate [6–8]. The nugget-to-sill ratio is therefore a very useful tool when assessing and comparing different process measurement systems.

Table 1. Important parameters for characterisation of the relative variogram [1,2,7,8,11].

Notation	Definition	Description
V(0)—Nugget effect	Extrapolated intersect of y-axis	Short range random variability stemming from the total measurement system, including sampling, sub-sampling, sample preparation and analytical measurement. The nugget effect is according to TOS the sum of all CSE and ISE, as well as the Total Analytical Error (TAE).
V(1)	Value at V(1)	The total variability occurring in the time between two samples, i.e., the total measurement system variability and the process variability that occurs between any two consecutive analyses.
V(Process)	V(1)–V(0)	Process variability for the time interval between two samples, not including the total measurement system variability. The relationship between V(process) and V(0) is an indication of the possibility to control the process with current sampling interval.
Sill	'Ceiling level' of the variogram	Describes the highest variability, or the global heterogeneity, in the data series. Technically the sill is the average of all variogram point values.
Range	Where the variogram reaches the sill	For process variograms the range can be directly translated into a time interval where paired samples show auto-correlation. The range is where an increasing variogram has flattened out.
Nugget-to-sill ratio	Relative measurement system variability	The nugget divided by the sill describe the variability stemming from the measurement system, relative to the overall variability. This ratio is an effective quality grading for any measurement system, also allowing distinct comparison between systems.

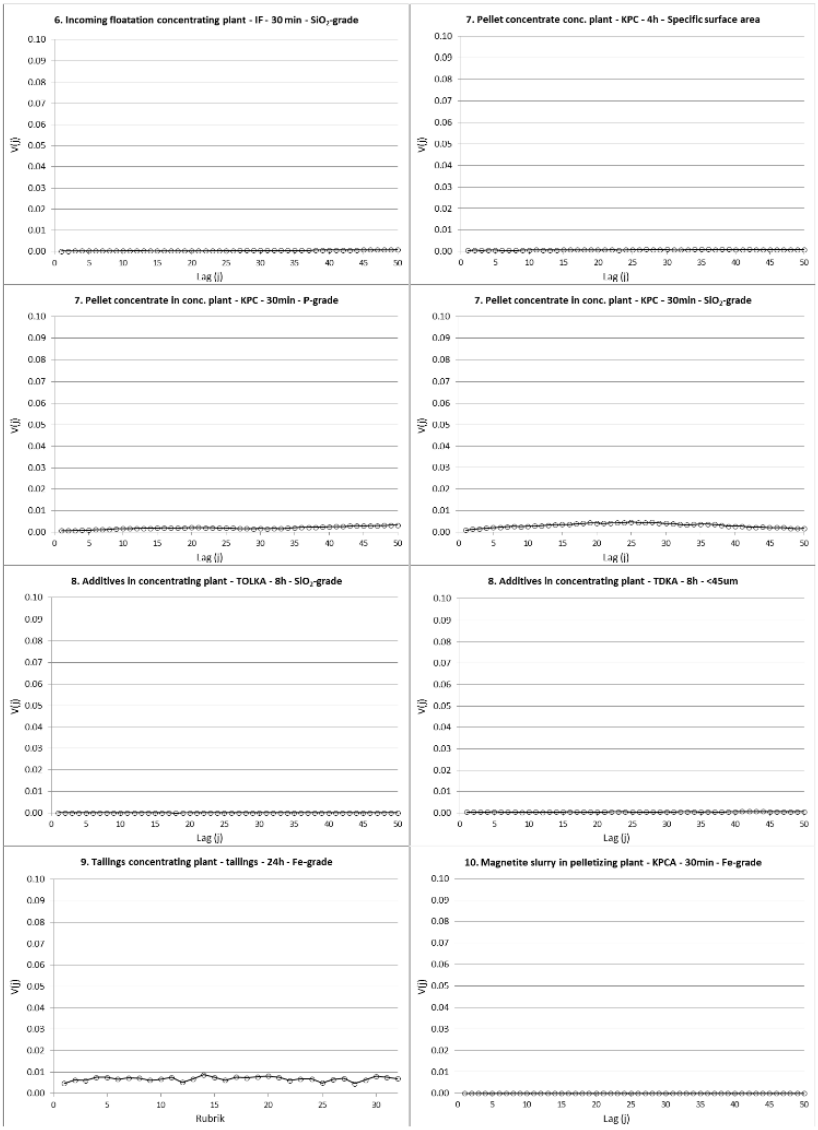
3. Results

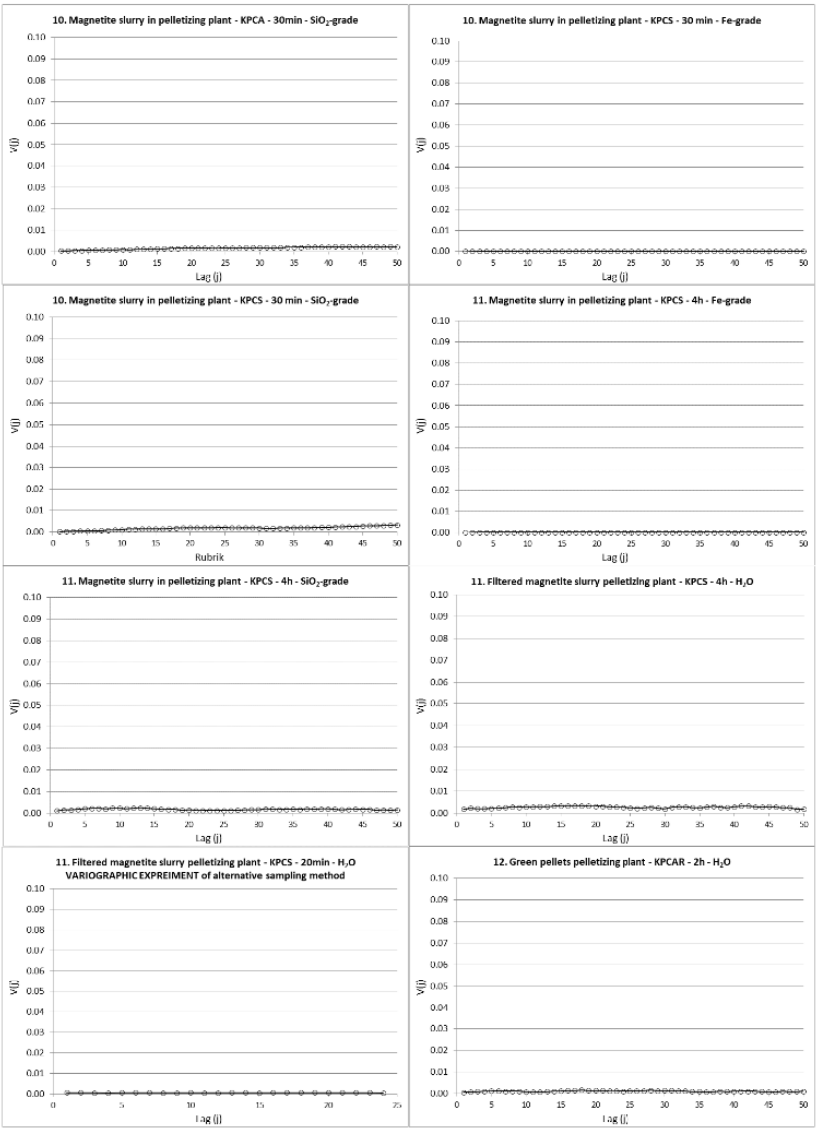
3.1. Variographic Characterization

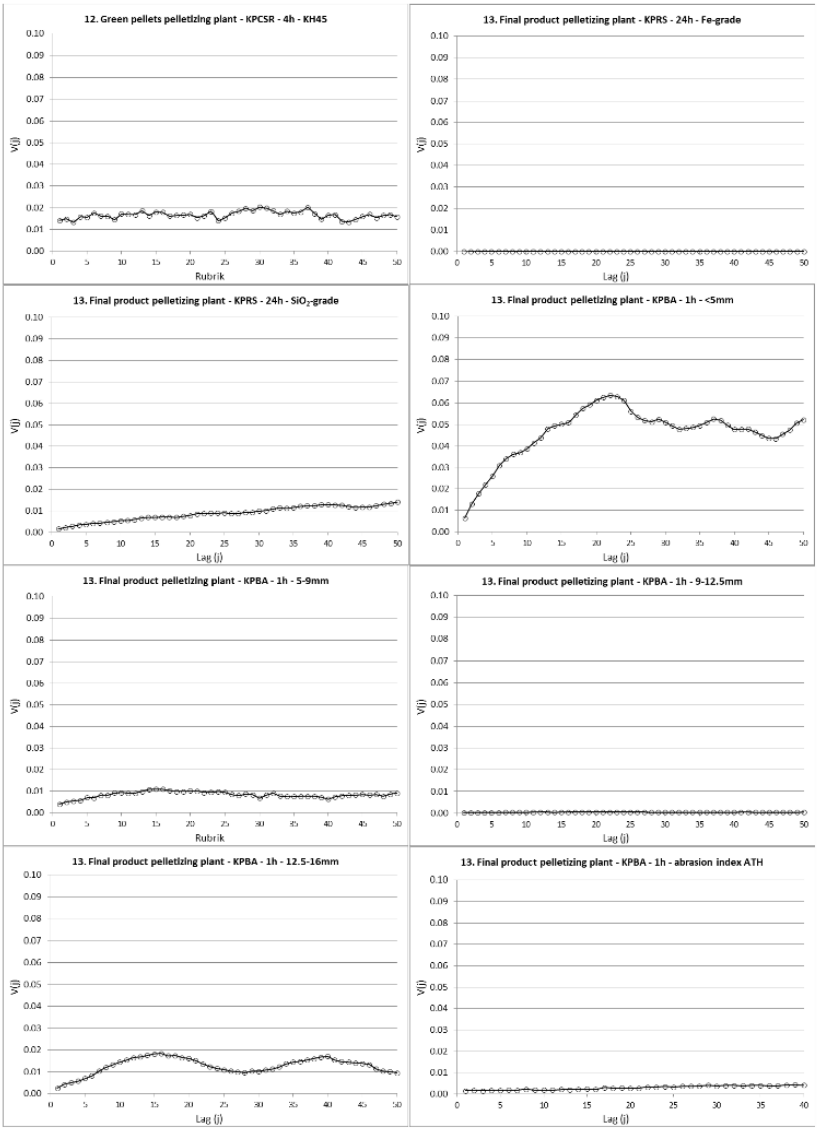
The variographic characterisation of the full iron ore processing value chain, at 14 sampling locations, see Figure 1, resulted in a total of 120 individual semi-variograms. For each sampling location and analytical parameter, two to four variograms were prepared, using 100 data points from separate process times. The purpose of generating replicated variograms for each measurement system was to enable assessment of the general performance for each measurement system over a selected, representative time interval for each measurement system. The objective was to cover six months of operation for measurement systems with up to one-hour analytical interval and one year of operation for measurement systems with more than one hour analytical interval. The selection was based on local process experiences, e.g., locating time periods with stable measurement system performance and eliminating time periods for maintenance shutdowns of the plants. Based hereupon this paper will only present one exemplar variogram for each measurement system, inferred to show the typical performance at each location. To enable a first comparison between the different measurement systems, all relative semi-variograms (except one) are presented on equal scaled y-axis, Figure 2. The choice of relevant lag units for each location/measurement system reflects the current sampling interval used at LKAB. The overview of all calculated variograms is presented below, as a first base-line characterisation of the critical process measurement systems.

The overview results show that the majority of the relative variograms exhibit a low total variability, below 0.01. For many measurement systems this is indicating that the specific processes are stable. However, detailed inspection of individual variograms reveal sampling locations with opportunity for improvements, i.e., where lower process variability and/or lower measurement variability, is needed. The nugget-to-sill ratio is used to quantify if the measurement systems are fit-for-purpose to describe the variability of the process monitored, i.e., if the measurement system variability is below 30% [6].


1. Incoming sorting plant - ore - 8h - Fe-grade
2. Final product sorting plant - AMD: 0-30mm - 8h - Fe-grade
2. Final product sorting plant - AMDF: 0-10mm - 8h - Fe-grade
3. Tailings sorting plant - 0-30mm - 8h - Fe-grade
3. Tailings sorting plant - 0-30mm - 5 min - Fe-grade
VARIOGRAPHIC EXPERIMENT
4. Incoming concentrating plant - AMDF - 0-10mm - 4h - Fe-grade
5. Fine fraction first milling concentrating plant - FK - 8h - Fe-grade
6. Incoming floatation concentrating plant - IF - 30 min - P-grade
v(j)
Lag (j)


6. Incoming floatation concentrating plant - IF - 30 min - SiO2-grade
7. Pellet concentrate conc. plant - KPC - 4h - Specific surface area
7. Pellet concentrate in conc. plant - KPC - 30min - P-grade
7. Pellet concentrate in conc. plant - KPC - 30min - SiO2-grade
8. Additives in concentrating plant - TOLKA - 8h - SiO2-grade
8. Additives in concentrating plant - TDKA - 8h - <45um
9. Tallings concentrating plant - tallings - 24h - Fe-grade
10. Magnetite slurry in pelletizing plant - KPCA - 30min - Fe-grade
V(j)
Lag (j)
Rubrik


10. Magnetite slurry in pelletizing plant - KPCA - 30min - SiO2-grade
10. Magnetite slurry in pelletizing plant - KPCS - 30 min - Fe-grade
10. Magnetite slurry in pelletizing plant - KPCS - 30 min - SiO2-grade
11. Magnetite slurry in pelletizing plant - KPCS - 4h - Fe-grade
11. Magnetite slurry in pelletizing plant - KPCS - 4h - SiO2-grade
11. Filtered magnetIte slurry pelletizing plant - KPCS - 4h - H2O
11. Filtered magnetite slurry pelletizing plant - KPCS - 20min - H2O
VARIOGRAPHIC EXPREIMENT of alternative sampling method
12. Green pellets pelletizing plant - KPCAR - 2h - H2O
V(j)
Lag (j)
Rubrik


12. Green pellets pelletizing plant - KPCSR - 4h - KH45
13. Final product pelletizing plant - KPRS - 24h - Fe-grade
13. Final product pelletizing plant - KPRS - 24h - SiO_2-grade
13. Final product pelletizing plant - KPBA - 1h - <5mm
13. Final product pelletizing plant - KPBA - 1h - 5-9mm
13. Final product pelletizing plant - KPBA - 1h - 9-12.5mm
13. Final product pelletizing plant - KPBA - 1h - 12.5-16mm
13. Final product pelletizing plant - KPBA - 1h - abrasion index ATH
V(j)
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Lag (j)

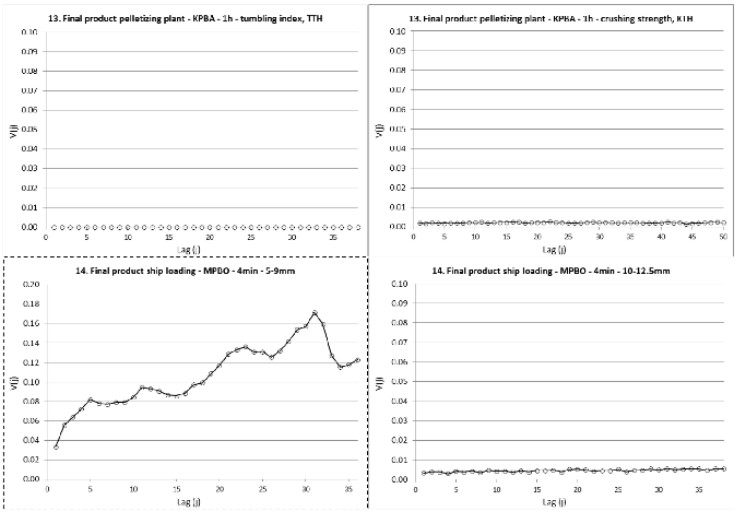

Figure 2. 36 relative variograms representing a total of 14 sampling locations and 34 measurement systems. Two specific variographic experiments are presented, one for a current measurement system (Fe-grade of tailings in the sorting plant) and one for an alternative measurement system (moisture content of filtered slurry in the pelletizing plant). The scale on the y-axis, $v(j)$, is equal on all variograms ($v(j)$ = 0–0.1) except one, marked with dashed black frame ($v(j)$ = 0–0.2).

3.1.1. High-Sill Variograms

Three relative variograms show significantly higher variabilities compared to all other, i.e., sill levels of ≈0.05, ≈0.07 and ≈0.14, respectively. Two of these three high-sill variograms are increasing variograms. These two variograms are describing the measurement systems 13: final product in pelletizing plant, 1 h analysis for <5 mm size fraction and sampling location 14: final product ship loading, 4 min analysis of 5–9 mm size fraction. The measurement systems for these two locations are equivalent, i.e., automated linear cross stream samplers, extracting increments every five and three minutes respectively. Both measurement systems further include automated rotary division and on-line sieving analysis. Both variograms exhibit a low nugget effect and low nugget-to-sill ratios of ≈10% and ≈14%, respectively. This show that these two measurement systems are fit-for-purpose to describe the large variability seen in the size fractions of the production and ship loading processes.

The third variogram showing a high sill level is at sampling location 3: tailings in the sorting plant, 8h analysis for iron-grade. This variogram is close to flat, indicating that a large fraction of the visible variability is stemming from the measurement system. The nugget-to-sill ratio is approximately 70%, showing that the measurement system is inadequate for describing the process variation for Fe-grade of the 0–30 mm tailings in the concentrating plant. To study this measurement system in more detail, a further experiment was executed, extracting and analysing primary increments every five minutes, Figure 3. This experiment shows a lower sill, consistent with that the time for the experiment covered only four hours compared to the process variogram covering 80 h of production. The nugget effect for the five-minute sampling interval is significantly lower, compared to the eight-hour interval for process sampling, which is fully as expected when the sampling interval is lowered. However, the nugget to sill ratio is now approximately 35% which indicate that even with this small sampling

interval, the measurement system is barely fit-for-purpose for evaluating the Fe-grade of tailings in the sorting plant.

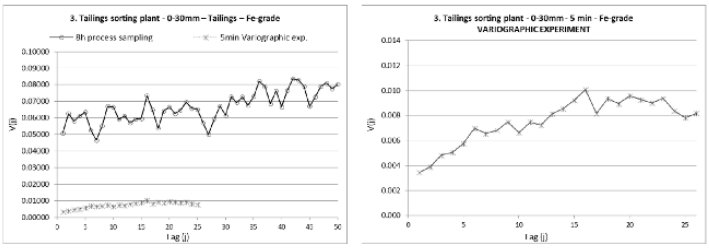

Figure 3. (**a**) Semi variogram for initial process sampling and variographic experiment conducted on sampling location 3, Fe-grade of tailings in the sorting plant (note the different time scales for the 8 h process sampling variogram and the 5 min variographic experiment). (**b**) Enlarged variogram for the 5-minute variographic experiment, conducted for Fe-grade of tailings in the sorting plant. Note the different scales of the y-axis in (**a**,**b**).

3.1.2. Medium-Sill Variograms

Four of the variograms indicate slightly higher overall variabilities of the low-sill types (Figure 4). These four show sill levels of ≈0.018, 0.012, 0.010 and 0.016. The morphology of these medium-sill variograms are significantly different.

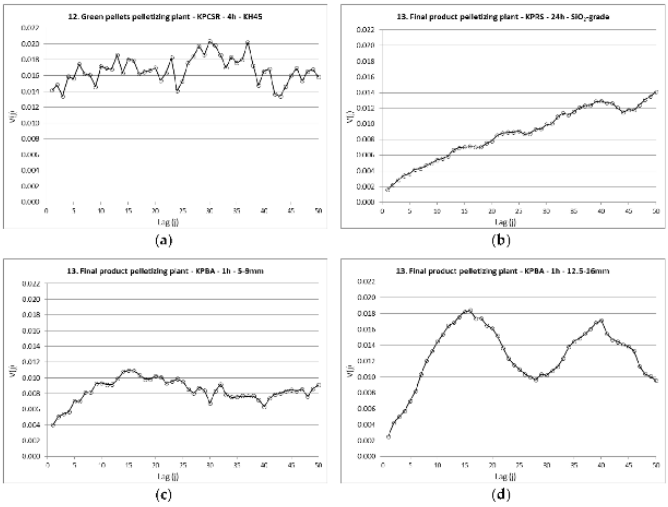

Figure 4. Relative variograms for the four measurement systems with sill levels of approximately 0.1–0.2%. (**a**) Variogram for measurement system 12:4 h KH45 analysis of green pellets, (**b**) Variogram for measurement system 13:24 h analysis of silica grade of DR pellets, (**c**) Variogram for measurement system 13:1 h analysis of size fraction 5–9 mm of BF pellets, (**d**) Variogram for measurement system 13:1 h analysis of the size fraction 12.5–16 mm of BF pellets.

Measurement system 12 (Figure 4a) 4h KH45 analysis of green pellets, is showing a typical flat variogram with a nugget-to-sill ratio of 81%. This measurement system is in itself responsible for the majority of variability observed. This means that the measurement system error is prohibiting a reliable visualization of the true process variability.

Measurement system 13 (Figure 4b) 24 h analysis of silica grade of the final product, DR pellets, shows a continuously increasing variogram, meaning that the autocorrelation between silica grade measurements is sustained throughout the complete process time available for the variogram. The nugget effect is low, and the nugget-to-sill ratio is 10%, showing that the measurement system is fit-for-purpose for determining silica grade of the DR pellets.

Measurement system 13 (Figure 4c) 1h analysis of size fraction 5–9 mm of the final product, BF pellets is showing an increasing variogram levelling off at range equivalent to lag ≈12. This is representing 12 process hours, indicating that there is no autocorrelation between samples spaced more than 12 h apart. The nugget-to-sill ratio of this measurement system is approximately 30% which is just acceptable.

Measurement system 13 (Figure 4d) 1h analysis of the size fraction 12.5–16 mm of BF pellets shows a typical periodic variogram. The period is approximately 26 lags representing just over 24 h production. The process sieving system, determining the size of green pellets going in to the sintering process is not a process parameter that is changeable from the control room, meaning that human factors cannot be the cause of the periodicity. This periodicity is only present in one of the prepared variograms for this measurement systems. The variability in this periodic variogram is higher (for both nugget effect and sill) compared to the other variograms for the same measurement system. There are apparently non-trivial variations in the process at this specific time period at this sampling location. The nugget-to-sill ratio for the three variograms for this measurement system (the two other variograms are increasing variograms with ranges of 25–30 lags) are all between 3–10%, representing a measurement system that is fit for process control purposes.

3.1.3. Measurement Systems with Distinct Variograms

The measurement system for moisture determination of filtered magnetite slurry in the pelletizing plants at LKAB, location 11: Filtered magnetite slurry in pelletizing plant, 4 h, moisture content, has previously been evaluated in Engström et al. [24]. The variographic characterization of the process sampling for moisture content of filtered slurry shows a nugget-to-sill ratio of approximately 60%, Figure 5. This shows that the measurement system is not fit for process control purposes, as it is not able to discern the relevant true process variability. Engström et al. [24] evaluated both the current process sampling method and two alternative measurement systems through variographic experiments, Figure 5. The results indicate that both on-line IR analysis as well as manual sampling of individual filters can produce significantly improved measurements with lower variability than the current process sampling method.

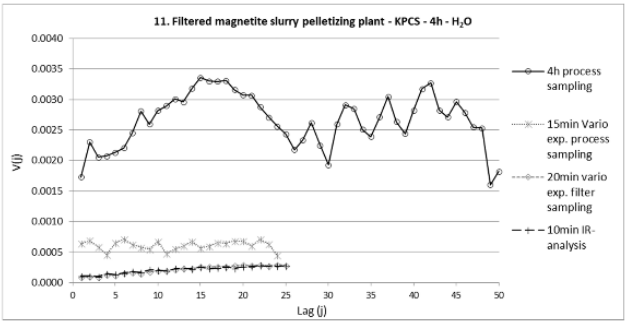

Figure 5. Semi variograms for the process sampling and three variographic experiments conducted on sampling location 12, moisture content of filtered magnetite slurry in the pelletizing plant (Variographic experiment results are previously presented in Engström et al. [24]. Note different time intervals for the separate variogram lags.

4. Discussion

4.1. Discrete Time Sampling for Variographic Characterization

Manual data collection was used in this study to extract data from the process control system and transfer the data to excel data sheets for variographic calculation. The excel calculation template for variogram development is originating from the standard DS3077 [6]; discrete time sampling must be performed to extract up to 100 equally time spaced data points from the process control system. In practice, any maintenance or disturbance in the measurement systems or maintenance shutdowns of processing plants can result in missing data and disruptions in the time spacing of data points. Such episodes must be eliminated from the possible time periods for valid variogram calculation. For each of the alternative variograms generated for the 34 measurement systems in this study, random discrete time sampling was performed within the normal operating process periods identified. The purpose of preparing several variograms for each measurement system was to evaluate the typical performance of each measurement system. For a few measurement systems, single variograms show deviations, e.g., periodicity, but for the majority, replicate variograms show similar appearances and nugget-to-sill ratios.

It should be noted that the lag distance for the different variograms presented is varying between four minutes for the incremental size determination for the ship loading of iron ore pellets, up to 24 h for example iron grade of tailings in the sorting plant and final product in the pelletizing plant. The lag distance is specified in the headline of each variogram. The reason for these large lag differences is that the evaluated measurement systems are the ones currently operating within LKAB, with the analytical intervals set in accordance to the specific process control requirements.

There are some unavoidable consequences of presenting a comparative overview of variograms based on disparate lag units. A consequence of a small lag spacing, e.g., five minutes, is that all process variability may not be included in the full-time interval covered by the variogram. This might lead to that some missed variability components or periodicities. On the other hand, a large lag spacing, e.g., 8 or 24 h, might well lead to a high degree of process variability being included in the nugget effect. However, this is also an interesting aspect of the current measurement system and its possibility to clearly describe the process variability. Either way, the present study is meant as a first base-line survey with which to compare when improving the indicated measurement systems.

To achieve continuous measurement system monitoring, automated data collection and variogram generation is essential. Target values and tolerance limits for the measurement system and process

variability also need to be defined. One result of the present study is a project focused on finding a systematic framework for on-line measurement system evaluation, using variographic characterization. Some desired features of this system concern the use of larger data sets (longer time intervals), and to enable effective handling of missing data. This may for example include weighing of data, in relation to proximity, to promote rapid detection of deviation in measurement system or process variability.

4.2. *Identified Improvement Opportunities*

The results from this study indicate that most of the measurement systems within the LKAB processing value chain have low sill levels, i.e., low combined process and measurement system variabilities. Even though several of the variograms show a nugget-to-sill ratio above 30%, the low total variability (low sill) mean that the process is under control. However, some of the measurement systems with high nugget-to-sill ratio does show a total variability that is too high for its intended purpose and therefore need to be improved. The measurement systems showing a higher sill in the variographic characterization are in need of improvements (either regarding measurement system, process control or both). The following discussion will present some suggested improvements for measurement systems identified to have too high variabilities.

4.2.1. 1. Incoming Sorting Plant—Ore—8 h—Fe-Grade

The measurement system for Fe-grade of the incoming ore to the sorting plant starts with a cross belt hammer sampler and automated sorting and secondary sampling in several repetitions, Figure 6. Even though the sill level is relatively low, the variogram shows a nugget-to-sill ratio of approximately 50%, indicating that this measurement system contributes to a large part of the overall variability. Cross belt samplers are known to introduce significant IDE and IEE as the primary sampling equipment cannot transect the complete depth of the cross section of the stream. There will always be some part of the material left on the bottom of the belt. Especially for material of large particle top size (as in this sampling situation) the finer material being segregated to the bottom of the belt is likely to be under-sampled.

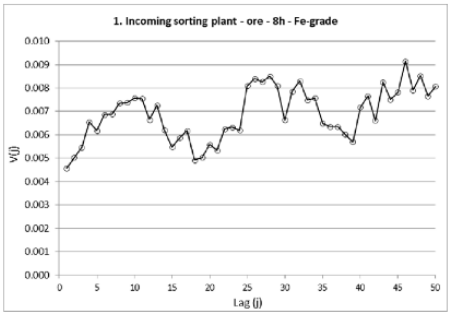

Figure 6. Relative semi-variogram for the measurement system of iron grade of the incoming ore to the sorting plant.

The variogram is also indicating some degree of periodicity, perhaps at two different time scales. This could for example be due to the variability from the raw material being mined in different parts of the mine. However, due to the distinct non-representative sample extraction method, this might also be due to periodic variabilities stemming from the measurement system.

A pilot study is initiated to evaluate the possibility of exchanging the hammer samplers at this sampling location and replace them with on-line analysers. The most interesting alternative being

evaluated at the moment is to use Pulsed fast and thermal neutron activation (PFTNA) technology. PFTNA use the energy of the gamma rays from the nuclei of the atoms that are induced by the neutrons to quantify the elements of the material. This technique is expected to allow improved analysis of iron grade, as well as nitrogen-grade which are important parameters for the refining processes. An advantage of the PFTNA technique, in contrast, many sensor- and on-line analysis methods can only analyse the surface, e.g., XRF and IR-analysis, is the possibility for complete penetration of the material, allowing analysis of the complete cross section of the material stream.

4.2.2. 3. Tailings Sorting Plant—0–30 mm—8 h—Fe-Grade

The variogram for iron grade of the tailings in the sorting plant is showing both a high sill and a high nugget-to-sill ratio of 70%, which shows that the measurement system is not fit for describing the true process variability. Analysis of iron grade of the tailings is a critical measurement for assessing the performance of the magnetic separation performed in the sorting plant. The primary sample extraction is done by manual sampling of the material that has a 30 mm top size. No matter how good the knowledge or intentions of the sampling staff, it is not possible to collect a representative sample using manual extraction at this location. As for the measurement system for Fe-grade of the incoming ore to the sorting plant (described above) a pilot study is currently evaluating the possibility of installing on-line analysis using PFTNA technique to improve this measurement system.

4.2.3. 5. Fine Fraction First Milling Concentrating Plant—FK—8 h—Fe-Grade

The analysis of iron grade of the fine fraction is used for reconciliation purposes in the concentrating plant, Figure 7. Even though the overall relative variability, described by the sill, is low, the high iron grade of the material does in practice mean that the absolute variability of the process control data is higher than desirable. Furthermore, the nugget-to-sill ratio of 75% is suggesting that most of the variability visible in the process control data is stemming from the measurement system rather than the process itself. The primary sample extraction for this measurement system is currently manual collection of slurry from the overflow of the spiral separator after primary milling. This sample extraction method disables any possibility for a representative cross section of the slurry material. A possible improvement would be to use a recirculation system where the sample extraction is placed in the smaller recirculating slurry flow. This would enable a more correct sample extraction as well as the possibility to collect a composite sample for analysis, in contrast to the current manual grab sampling.

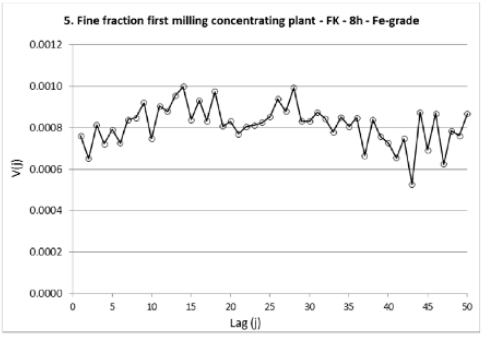

Figure 7. Relative semi-variogram for the measurement system of iron grade of the fine fraction from the first milling in the concentrating plant.

4.2.4. 11. Filtered Magnetite Slurry Pelletizing Plant—KPCS—4 h—H_2O

The moisture sampling of filtered magnetite slurry has previously been studied in Engström et al. (2018). The conclusion was that the current belt sampling method does not achieve representative measurements and the measurement system need to be improved. The present study shows similar results, where the nugget to sill ratio is approximately 50% and the moisture content requirements are so precise that a lower variability in both process and measurement is necessary. As correct moisture content of filtered slurry is a critical aspect for successful balling of green pellets, this is a measurement system improvement priority. A current pilot study is evaluating the possibility to use machine learning to predict the moisture content of each individual filter, based on the abundant availability of process data. Parallel to this, both infra-red and image analysis are evaluated and tested to determine if any of these on-line methods could replace the current non-representative manual grab sampling.

4.2.5. 12. Green Pellets Pelletizing Plant—KPCSR—4 h—KH45

For the measurement system of 4h analysis of the analytical parameter KH45, the primary sampling method is again a manual grab sample. The variogram is indicating a higher sill compared to most other measurement systems, and the nugget-to-sill ratio of approximately 75% is suggesting that the true process variability is largely masked by the measurement system variability. The method of sample extraction is not conforming to TOS, and furthermore, the analytical method is a mechanic and operator dependent, leading to large variabilities in both sampling and analysis. As the green pellets are quite fragile, automated sampling or splitting is not possible to apply to this product, leaving small possibilities for improvement. Internal studies at LKAB have also indicated large measurement uncertainty in the KH45 analysis and the purpose of its usefulness is therefore questionable. Perhaps it is better not to measure when conditions for proper sampling and analysis are so adverse?

4.2.6. 13. Final Product Pelletizing Plant—KPBA—24 h—SiO_2-Grade

The analysis of silica grade for the final product in the LKAB value chain, high quality iron ore pellets, stand out with a slightly higher sill (0.012) than most other presented variograms. However, the variogram is indicating a low nugget effect and therefore the measurement system can be deemed fit for its quality control purposes, with a nugget-to-sill ratio of under 10%. The main reason for the high sill is that the variograms are calculated based on relative heterogeneity contributions and as the silica grade is so low in the final product, the relative process variability for silica is higher than for iron grade.

4.2.7. 13. Final Product Pelletizing Plant—KPBA—1 h—Size Determination

For three of the presented size fractions, the relative variograms indicate a higher sill compared to most other measurement systems. The sill levels for <5 mm, 5–9 mm and 12.5–16 mm are approximately 0.05, 0.01 and 0.016 respectively. The nugget-to-sill ratios are however all under 30% and both <5 mm and 12.5–16 mm have ratios under 10%. This is indicating that even though the relative process variability is high at this sampling location, the measurement system is fit-for-purpose for accurate quality control. The highest relative process variability for <5mm is also largely due to the low mean value (0.8%) of this size fraction. As this measurement system is evaluating the final product in the pelletizing plant, the analysis is used for quality assessment rather than for process control. All size determinations of the final product are determined on the same samples with the same analytical method; the fact that the variogram range for all three parameters are identical (12 h) is consistent.

4.2.8. 14. Final Product Ship Loading—MPBO—4 min—Size Determination

Figure 8a–c shows three replicated high resolution variograms representing three separate ship loading operations for the 5–9 mm size fraction of BF pellets. The variogram for the ship loadings are based on a high-resolution study of the individual size determinations made on each primary

increment extracted with four-minute intervals. The final analytical result used for product certification is based on composite averages of all 75–115 size determinations for a fully loaded ship, leading to a highly reliable product specification.

The high resolution variograms were prepared in order to characterize the loading measurement system specifically. At this high resolution, the variability (sill level) is necessarily high, due to individual increment analysis uncertainty, but these variograms are only used to characterize the nugget effect. Two of the replicates (Figure 8a,b) show similar variograms, while the third (Figure 8c) has a distinctly higher sill and nugget effect, reflecting minor differences in the heterogeneity of the loaded material, which is substantiated by slight differences in the mean values for the size fraction (proprietary information). The three nugget effects are broadly similar (0.03, 0.03, 0.05), indicating a measurement system fit-for-purpose, which is justified by the three nugget-to-sill ratios: 20%, 20%, 14%.

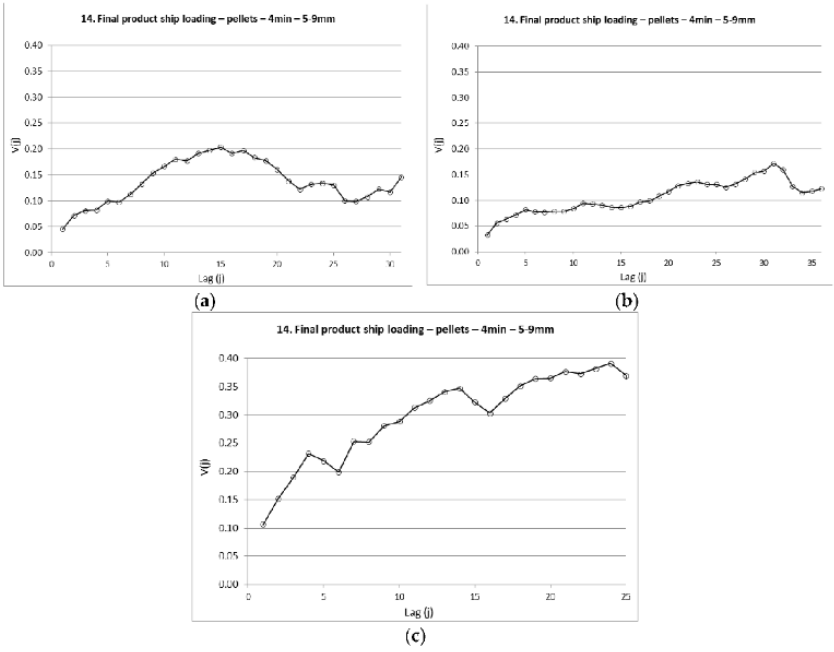

Figure 8. Three replicated high resolution variograms for separate ship loadings, for individual increment determination of the size fraction 5–9 mm. The high resolution variograms were prepared in order to characterize the loading measurement system in full detail.

4.3. Slurry Sampling Issues

Slurry sampling is notoriously difficult, due to the limited possibility to access the material streams in long and/or pressurised pipelines. This study found interesting results related to the performance of TOS incorrect slurry shark-fin and spear sampling. Several measurement systems at LKAB uses these sampling approaches in slurry pipes and blender tanks. Conventional TOS wisdom claims that these are inferior sampling methods, but the present study revealed contradictory results, related to relative uniform materials.

The primary sampling method for phosphorus grade of incoming floatation (IF) is a spear sampler applied to a pressurized slurry pipe. The primary sample is divided in several secondary sampling steps, milled and analysed by automated XRF analysis every 30 min. The variographic characterisation, Figure 9, show an increasing variogram with low nugget effect and a nugget-to-sill ratio of 25%. This is an acceptable level for the difficult case of slurry sampling of pressurized pipes.

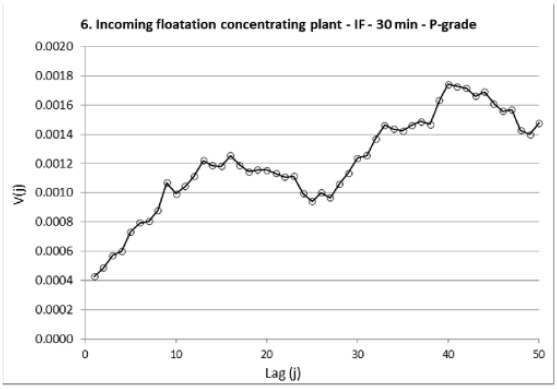

Figure 9. Relative variogram for the phosphorus grade determination of incoming floatation, based on pressurized spear primary sampling.

The measurement system for silica grade of pellet concentrate applies a shark-fin slurry sampler for primary sample extraction, followed by equivalent sample preparation and analysis as the phosphorus determination previously described. The variographic characterization, Figure 10, also shows a satisfactory nugget effect and a nugget-to-sill ratio of 20%, acceptable for the shark-fin case. The results in Figures 9 and 10 are likely related to the relatively uniform material being sampled. This empirical study thereby validates the current slurry measurement systems used by LKAB, but the results cannot be readily applied to slurry samplers in general.

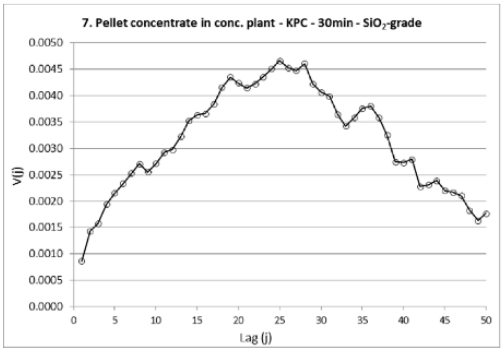

Figure 10. Relative variogram for the silica grade determination of pellet concentrate, based on slurry shark-fin primary sampling.

The measurement system for silica grade of magnetite slurry applies primary spear sample extraction from a blender tank. The measurement system is recirculating the extracted primary sample back to the blender tank, while it is sub-sampled in subsequent stages (using TOS correct cross stream secondary sampling) to reach the final analytical aliquot. The variogram shows a low nugget effect and a nugget-to-sill ratio of 10%. This is an indication that the slurry material in the blender tank is sufficiently well mixed when being subject to the primary sample extraction. This correspond to the experiences of process and research engineers, confirming that with correct slurry level in the tank, the slurry mixing is adequate and not subjected to segregation. The characterization of this measurement system shows an acceptable measurement system performance.

The three measurement systems characterized in Figures 9–11 pertain to a 30 min lag interval, corresponding to automated sample preparation and XRF analysis of the divided primary samples. This allow for detailed characterization of the measurement systems in use. The measurement system for silica grade and <45 um of additives are in contrast based on 8h off-line laboratory XRF and sieving analysis respectively. Both these apply shark-fin primary sampling collecting a part of the slurry stream all of the time. The primary sample is aggregated to 8h composite samples brought to the laboratory for analysis.

The variographic characterisation of these two measurement systems, Figure 12, shows flat variograms with a nugget-to-sill ratio close to 100%. The reason for this is likely due to that the grinding process preceding the measurement systems is a 'stop and go' process. The grinding circuit is operated towards the filling level of the additive tank. When the tank is full, the grinding is halted, and only started again when material is needed. This results in large material variations when the grinding circuit is emptied and started, with large variations in starting and finishing phases. As this situation often occur within the 8 h sampling interval, the full variation in the process is found within one sampling interval, leading to the observed flat variograms. Periodic variographic characterisation, i.e., updating a variogram at process- and material dependent intervals, is a powerful on-line process/product quality control tool. Monitoring the process and providing an on-line performance grading (nugget effect-to-sill ratio) of the measurement system.

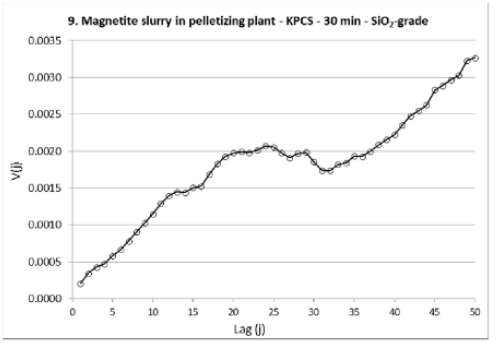

Figure 11. Relative variogram for the silica grade determination of magnetite slurry, based on primary spear sampling from a blender tank.

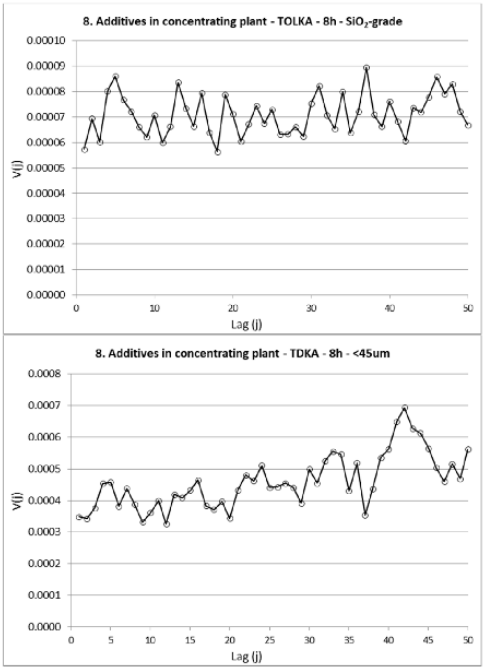

Figure 12. Relative variograms for the measurement systems for silica grade and <45 um of additives, based on 8 h laboratory XRF and sieving analysis respectively. Both these systems use shark-fin samplers for primary sample extraction.

5. Conclusions

Variographic characterisation of 14 critical sampling locations, covering a total of 34 separate measurement systems, show significant disparity along the full processing value chain at LKAB, Sweden. A majority show low sill, i.e., low overall variability which means that many part processes are well controlled, and many variograms also show low nugget effect, indicating satisfactory measurement systems. The notable exceptions point to measurement systems in need of improvements. Where only some were previously known with certainty, variographic analysis now opens up for objective discussion between professions. This study substantiates the general TOS understanding that manual sampling and cross belt hammer samplers is leading to unacceptably large sampling errors and should be abandoned. However, it is also found that slurry shark-fin and spear sampling show acceptable variogram characteristics for these types of material, in contradiction to the general expectations within TOS. This is underlining the strength of comprehensive empirical studies.

The most important aspect of this study is the possibility to characterize process measurement systems statistically with the powerful variographic technique. The present comprehensive compilation of variographic results, covering a full processing value chain, represent a new approach at LKAB. On this basis, it is now possible to conduct rational enquiry of all measurement systems, enabling objective prioritization of where improvement efforts may have the largest cost-benefit effect. After this first base-line survey, more focused variographic studies will for example involve finer resolution details and alternative sampling methods. Variographic characterization can also provide a quality control

tool for continuous evaluation of measurement system performance (periodic updating of on-line variograms). Furthermore, variographic characterization is superior for comparing before-and-after measurement system replacement, which will be a natural element in company improvement projects. Optimised process measurement systems will have a positive effect on the company bottom line, i.e., less wasted material, more effective processing, fewer hidden costs and improved decision making.

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